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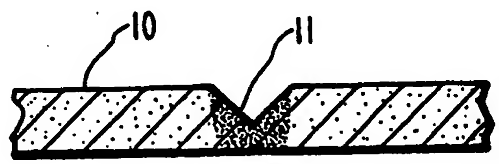
(54) Title: HINGES FOR HYDRAULICALLY SETTABLE MATERIALS

(57) Abstract

A hinge (11) for use in hydraulically settable materials is provided. The hinge (11) has a hydraulically settable matrix (10) formed from a reaction product of a hydraulically settable binder and water, with fibrous material dispersed in the matrix. Additional components may be utilized in the matrix (10), such as rheology-modifying agents, aggregate materials and substances to produce air voids. The hinge (11) of the invention allows hydraulically settable materials to be bent along a line without breakage of the material. The hinge (11) is particularly useful in cementitious containers that require bending of various container parts, such as in food containers and boxes made from cementitious materials.



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HINGES FOR HYDRAULICALLY SETTABLE MATERIALS

BACKGROUND

1. Field of the Invention

The invention relates generally to a hinge for use with hydraulically settable materials. More particularly, the invention relates to a hinge integrally made in a hydraulically settable sheet such as a cementitious sheet, which can be formed into various containers or other products.

2. Related Applications

This application is a continuation-in-part of co-pending application Serial No. 08/101,500 entitled "Methods and Apparatus for Manufacturing Moldable Hydraulically Settable Sheets Used in Making Containers, Printed Materials, and Other Objects," filed August 3, 1993 in the names of Per Just Andersen, Ph.D. and Simon K. Hodson. This application is also a continuation-in-part of co-pending application Serial No. 08/095,662 entitled "Hydraulically Settable Containers and Other Articles for Storing, Dispensing, and Packaging Food and Beverages and Methods for Their Manufacture," filed July 21, 1993 in the names of Per Just Andersen, Ph.D. and Simon K. Hodson. This application is also a continuation-in-part of co-pending application Serial No. 08/019,151 entitled "Cementitious Materials For Use In Packaging Containers and Their Methods of Manufacture," filed February 17, 1993 in the names of Per Just Andersen, Ph.D. and Simon K. Hodson. This application is also a continuation-in-part of co-pending application Serial No. 07/929,898 entitled "Cementitious Food and Beverage Storage, Dispensing, and Packaging Containers and The Methods of Manufacturing Same," filed August 11, 1992 in the names of Per Just Andersen, Ph.D. and Simon K. Hodson.

3. The Relevant Technology

A. Traditional Hydraulically Settable Materials

Hydraulically settable materials such as those that contain hydraulic cement or gypsum (hereinafter "hydraulically settable," "hydraulic," or "cementitious" compositions, materials, or mixtures) have been used to create useful, generally large, bulky structures that are durable, strong, and relatively inexpensive. For example, cement is a hydraulically settable binder derived from clay and limestone, and it is essentially nondepletable.

Those materials containing a hydraulic cement are generally formed by mixing hydraulic cement with water and usually some type of aggregate to form a cementitious mixture, which hardens into concrete. Ideally, a freshly mixed cementitious mixture is fairly nonviscous, semi-fluid, and capable of being mixed and formed by hand. Because of its fluid nature, concrete is generally shaped by being poured into a mold, worked to eliminate large air pockets, and allowed to harden. If the surface of the concrete structure is to be exposed, such as on a concrete sidewalk, additional efforts are made to finish the surface to make it more functional and to give it the desired surface characteristics.

Due to the high level of fluidity required for typical cementitious mixtures to have adequate workability, the uses of concrete and other hydraulically settable mixtures have been limited mainly to simple shapes which are generally large, heavy, and bulky, and which require mechanical forces to retain their shape for an extended period of time until sufficient hardening of the material has occurred. Another aspect of the limitations of traditional cementitious mixtures or slurries is that they have little or no form stability and they are molded into final form by pouring the mixture into a space having externally supported boundaries or walls.

It is precisely because of this lack of moldability (which may be the result of poor workability and/or poor form stability), coupled with the low tensile strength per unit weight, that hydraulically settable materials have traditionally been useful only for applications where size and weight are not limiting factors and where the forces or loads exerted on the concrete are generally limited to compressive forces or loads, as in, *e.g.*, roads, foundations, sidewalks, and walls.

Moreover, previous hydraulically settable materials have been brittle, rigid. unable to be folded or bent, and have low elasticity, deflection and flexural strength. The brittle nature and lack of tensile strength (about 1-4 Mpa) in concrete is ubiquitously illustrated by the fact that concrete readily cracks or fractures upon the slightest amount of shrinkage or bending, unlike other materials such as metal, paper, plastic, or ceramic. Consequently, typical hydraulically settable materials have not been suitable for making small, lightweight objects, such as containers or thin sheets, which are better if made from materials with much higher tensile and flexural strengths per unit weight compared to typical hydraulically settable materials.

More recently, higher strength hydraulically settable materials have been developed which might be capable of being formed into smaller, denser objects. One such material is known as "Macro-defect Free" or "MDF" concrete, such as is disclosed in U.S. Patent No. 4,410,366 to Birchall et al. *See also*, S.J. Weiss, E.M. Gartner & S.W.

Tresouthick, "High Tensile Cement Pastes as a Low Energy Substitute for Metals, Plastics, Ceramics, and Wood," U.S. Department of Energy CTL Project CR7851-4330 (Final Report, November 1984). Such high strength cementitious materials have been prohibitively expensive and would be unsuitable for making inexpensive sheets or containers where much cheaper materials better suited for such uses (*e.g.*, paper and plastic) are readily available. Another drawback is that MDF concrete cannot be used to mass produce small lightweight objects due to the high amount of time and effort involved in forming and hardening the material and the fact that it is highly water soluble. Therefore, MDF concrete has been limited to expensive objects of simple shape.

Another problem with traditional, and even more recently developed high strength concretes has been the lengthy curing times almost universally required for most concretes. Typical concrete products formed from a flowable mixture require a hardening period of 10-24 hours before the concrete is mechanically self-supporting, and upwards of a month before the concrete reaches a substantial amount of its maximum strength. Extreme care has had to be used to avoid moving the hydraulically settable articles until they have obtained sufficient strength to be demolded. Movement or demolding prior to this time has usually resulted in cracks and flaws in the hydraulically settable structural matrix. Once self-supporting, the object could be demolded, although it has not typically attained the majority of its ultimate strength until days or even weeks later.

Since the molds used in forming hydraulically settable objects are generally reused in the production of concrete products and a substantial period of time is required for even minimal curing of the concrete, it has been difficult to economically and commercially mass produce hydraulically settable objects. Although zero slump concrete has been used to produce large, bulky objects (such as molded slabs, large pipes, or bricks which are immediately self-supporting) on an economically commercial scale, such production is only useful in producing objects at a rate of a few thousand per day. Such compositions and methods cannot be used to mass produce small, thin-walled objects at a rate of thousands per hour.

Demolding a hydraulically settable object can create further problems. As concrete cures, it tends to bond to the forms unless expensive releasing agents are used. It is often necessary to wedge the forms loose to remove them. Such wedging, if not done properly and carefully each time, often results in cracking or breakage around the edges of the structure. This problem further limits the ability to make thin-walled

hydraulically settable articles or shapes other than flat slabs, particularly in any type of a commercial mass production.

If the bond between the outer wall of the molded hydraulically settable article and the mold is greater than the internal cohesive or tensile strengths of the molded article, removal of the mold will likely break the relatively weak walls or other structural features of the molded article. Hence, traditional hydraulically settable objects must be large in volume, as well as extraordinarily simple in shape, in order to avoid breakage during demolding (unless expensive releasing agents and other precautions are used).

Typical processing techniques of concrete also require that it be properly consolidated after it is placed in order to ensure that no voids exist between the forms or in the structural matrix. This is usually accomplished through various methods of vibration or poking. The problem with consolidating, however, is that extensive overvibration of the concrete after it has been placed can result in segregation or bleeding of the concrete.

"Bleeding" is the migration of water to the top surface of freshly placed concrete caused by the settling of the heavier aggregate. Excessive bleeding increases the water to cement ratio near the top surface of the concrete slab, which correspondingly weakens and reduces the durability of the surface of the slab. The overworking of concrete during the finishing process not only brings an excess of water to the surface, but also fine material, thereby resulting in subsequent surface defects.

For each of the foregoing reasons, as well as numerous others which cannot be listed here, hydraulically settable materials have not generally had application outside of the formation of large, slab-like objects, such as in buildings, foundations, walk-ways, or highways, or as mortar to adhere bricks or cured concrete blocks. It is completely counterintuitive, as well as contrary to human experience, to even imagine (let alone actually experience) the manufacture from hydraulically settable materials of small lightweight sheets and other objects such as containers, which are comparable to light-weight sheets made from paper, cardboard, plastic, or polystyrene.

Due to the more recent awareness of the tremendous environmental impact (not to mention the ever mounting political pressures) of using sheets made from paper, card-board, plastic, polystyrene, and metals for a variety of single-use, mainly disposable, items such as containers or magazines, there has been an acute need (long since recognized to those skilled in the art) to find environmentally sound substitute materials.

In spite of such pressures and long-felt need, the technology simply has not existed for the economic and feasible production of hydraulically settable materials that

could be substituted for paper, cardboard, plastic, polystyrene, or metal sheets used to make a wide variety of disposable items such as containers. However, because hydraulically settable materials essentially comprise such environmentally neutral components such as rock, sand, clay, and water, they would be ideally suited from an ecological standpoint to replace paper, cardboard, plastic, or polystyrene materials as the material of choice for such applications.

B. The Impact of Paper, Cardboard, Plastic, Polystyrene, and Metals.

A huge variety of objects such as containers, packing materials, mats, disposable utensils, reading or other printed materials, and decorative items are presently mass-produced from paper, cardboard, plastic, polystyrene, and metals. The vast majority of such items eventually wind up within our ever diminishing landfills, or worse, are scattered on the ground or dumped into bodies of water as litter. Because plastic and polystyrene are essentially nonbiodegradable, they persist within the land and water as unsightly, value diminishing, and (in some cases) toxic foreign materials. Even paper or cardboard, believed by many to be biodegradable, can persist for years, even decades, within landfills where they are shielded from air, light, and water, which are all necessary for normal biodegradation activities. Metal products utilize valuable natural resources in their manufacture, and if not recycled, remain in the landfill and are unusable essentially forever.

Recently, with the public's attention being focused on environmental issues, certain containment products have come under heavy scrutiny, especially disposable packing materials and boxes. Most notably subject to criticism have been styrofoam products, which typically require the use of chloro-fluorocarbons (or "CFC's") in their manufacture, as well as use of vast amounts of the ever shrinking petroleum reserves. In the manufacture of foams, including styrofoam (or blown polystyrene), CFC's (which are highly volatile liquids) are used to "puff" or "blow" the polystyrene which is then molded into foam cups and other food containers or packing materials. Unfortunately, CFC's have been linked to the destruction of the ozone layer, because they release chlorine products into the stratosphere.

As a result, there has been widespread clamor for companies to return to using more environmentally safe and low cost containers. Some environmentalists have even favored a return to more extensive use of paper products instead of polystyrene, if only because it is thought by some that paper represents the lesser of two evils. Nevertheless, although paper products have not been linked to the destruction of the ozone layer and

are biodegradable, recent studies have shown that paper more strongly impacts the environment than does styrofoam in other respects.

In the manufacture of paper, the fiber slurry has upwards of 99% water which must be removed during the paper-making process. The slurry is sprayed onto a moving sieve bed through which water is extracted by a series of suction boxes. When the fiber sheet which is formed is removed from the moving bed, the fiber sheet still comprises 80% water. The fiber sheet then passes through a series of rollers which reduces the water content to about 50%; thereafter, heat is applied to dry the fiber sheet to form the paper product. This process, which has changed little in decades, is energy intensive, time consuming, and requires a significant initial investment.

Further, it is often necessary to coat many paper containers with a wax or plastic material in order to give it waterproofing properties. Moreover, if insulative properties are necessary, even more drastic modifications to the paper material in the container are necessary. Many types of plastic containers as well as coatings utilized with paper containers are derived from fossil fuels, mainly petroleum, and share many of the environmental concerns of petroleum refinement.

The manufacturing processes of plastic sheets or products vary, but they typically require precise control of both temperature and shear stress in order to make a usable product. In addition, the typical polystyrene or plastic manufacturing process is a high consumer of energy. Similarly, manufacturing products from metals consumes high amounts of energy because of the elevated temperatures utilized in the processes, as well as requiring high shear stresses to fashion and mold the products. Of course, the initial capital investments for manufacturing processes utilizing metals are very high.

About the only effective way to reduce the sheer volume of traditional container and packing wastes is through recycling. However, recycling is not without its contribution of large amounts of pollution into the environment in the form of fuel spent in transporting recyclables to recycling centers, as well as fuels and chemicals used in the recycling process itself.

In spite of the more recent attention that has been given to reduce the use of the above materials, they continue to be used because of their superior properties of strength and, especially, mass productivity. Moreover, for any given use for which they were designed, such materials are relatively inexpensive, lightweight, easy to mold, strong, durable, and resistant to degradation during the use of the object in question.

Although each of these materials may be comparably priced to any of the other materials presently available, they are usually far more expensive than typical

hydraulically settable materials. Because no rational business would ignore the economic benefit that would necessarily accrue from the substitution of radically cheaper hydraulically settable materials for paper, cardboard, plastic, polystyrene, or metal materials, the failure to do so can only be explained by a marked absence of available technology to make the switch.

Recently, there has been a growing debate as to which of these materials (e.g., paper, plastic or metals) is more damaging to the environment. Consciousness-raising organizations have convinced many people to substitute one material for another in order to be more environmentally "correct." The debate often misses the point that each of these materials has its own unique environmental weaknesses. To one who is not fully informed, or who may lack an adequately rigorous scientific approach, one material may appear superior to another when viewed in light of a particular environmental problem, while ignoring different, often larger problems associated with the supposedly preferred material.

In fact, paper, cardboard, plastic, polystyrene, and metal each has its own unique environmental weaknesses. The debate should, therefore, not be directed to which of these materials is more or less harmful to the environment, but should rather be directed toward asking: Can we find an alternative material that will solve most, if not all, of the various environmental problems associated with each of these presently used materials?

Based on the forgoing, what is needed are improved compositions and methods for manufacturing cementitious and other hydraulically settable sheets that can be formed into a variety of objects presently formed from paper, cardboard, polystyrene, plastic, or metal. To address these and other concerns, we have developed containers having a hydraulically settable structural matrix such as cementitious containers, which have numerous advantages over paper and styrofoam based containers found in the prior art.

Many containers, which can be formed without the need for any bending or folding, are readily adaptable to be manufactured from cementitious materials. These include plates, cups, utensils, etc. Many other types of containers such as boxes, clamshells, etc., however, require a material that can be bent and/or folded to form the desired shape and still be competitive in cost to manufacture. Accordingly, what is needed is a hinge adapted for use with a hydraulically settable material and, more particularly, a hinge that can be integrally formed as part of a sheet of hydraulically settable material, such as a cementitious material, that permits the sheet to be bent or folded into various configurations to form a variety of types of containers.

Hinges known as "living hinges" have been used in the past on various plastic molded products. A living hinge may be bent several times without breakage or fracture of the material. Living hinges have been formed from soft, flexible thermoplastic elastomers that exhibit high endurance to flexural fatigue. Living hinges can take various shapes and have been used on various plastic molded parts to provide pivotal movement between adjacent rigid parts.

Scoring is a technique that has been used to provide memory to sheet materials, such as paper-based materials, so that they bend in the same place along the scoring line. These materials are bent toward the score . Scoring of a paper-based material damages the fibers at the score, making the material weaker in the area of the score, which provides for the bending of the material along the score . Scoring has been used on various products such as on cardboard boxes to provide bendable flaps to close the box, foldable game boards, file folders, etc.

Scoring a hydraulically settable material to produce a hinge for bending of the material has not been heretofore possible since such a material was previously too thick or too brittle to provide an effective bending point without breaking.

Therefore, there is a need for a hinge for hydraulically settable materials that is at least as good as hinges used on prior paper or plastic products in order to produce various containers having easily bendable portions. Such a hinge is disclosed and claimed herein.

SUMMARY OF THE INVENTION

The present invention is an apparatus comprising a hinge formed of a hydraulically settable matrix and a fibrous material within the matrix at the hinge location. The hydraulically settable matrix comprises the chemical reaction products of a hydraulically settable matrix binder and water. The hydraulically settable matrix forming the hinge can further include other components such as a rheology-modifying agent and various aggregate materials.

The invention also includes an apparatus comprising a first member, a second member adjacent to the first member, and means for flexibly joining the first and second members so that the first and second members can be pivotally moved about the joining means relative to one another. The joining means has a hydraulically settable structural matrix including the reaction products of a hydraulically settable mixture comprising a hydraulically settable binder such as cement, fibers, and water. The joining means allows the first and second members to be pivotally moved between a first position wherein the

first and second members are in straight alignment with one another and a plurality of other positions wherein the first and second members form an angle in relation to one another.

The hinge of the present invention can be made in hydraulically settable sheets having properties similar to those of paper, plastic, or thin-walled metals. Such sheets can be immediately used to form a variety of objects such as food or beverage containers, or can be stacked or rolled and stored for future use. Stored sheets can be remoistened in order to introduce additional flexibility and elongation therein to avoid splitting or cracking when an object is formed.

The hinge of the invention can be advantageously formed during the sheet manufacturing process by scoring, creping or perforating a formed hydraulically settable sheet, which aids in forming a bend or hinge at a predetermined location within the sheet. The score can be cut or pressed into the surface of the sheet anytime after the sheet is formed in order to create a line within the structural matrix upon which the sheet can later be bent. Thus, the score can be molded between two parts of a mold, pressed into the sheet while in the green state or in a semi-hardened state, or the score can be cut into the sheet after the sheet has become fully dried. For example, a flat sheet can be scored and formed into the shape of a container and then hardened, or can be allowed to harden and then scored and formed into the shape of a container. The time and location of the placement of a score, score cut, or perforation will depend upon the desired purpose of the score and the properties of the hydraulically settable material in question.

The sheet preferably bends away from the score which is different from paper-based materials that bend toward the score. Furthermore, the hinge area of the hydraulically settable sheet at the score actually becomes stronger as a result of the densification of the hydraulically settable material at the score .

In addition, coatings can be applied to the surface of the sheet to make the sheet more flexible and can be applied to permanently enhance the flexibility or elastic modulus of the sheet or hinge within the sheet. Elastomer, plastic, or paper coatings can aid in preserving the integrity of the hinge whether or not the underlying hardened structural matrix fractures upon bending at the hinge.

During the subsequent process of forming the sheet into the shape of the desired object, it will usually be advantageous to remoisten the hardened sheet in order to temporarily increase the flexibility and workability of the sheet. This is particularly true in the case where the sheet will be rolled or has been scored and is expected to make a particularly sharp bend during the container forming stage.

The hinge of the invention may be used in a variety of containers such as boxes, clamshell containers, etc. The hinge of the invention formed by scoring can have a cross section with a variety of shapes such as a square, parabolic, sinusoidal, wedge, triangular shape, etc. Multiple scores may be used in order to provide increased bendability of the sheet without breaking or fracturing thereof.

Scoring allows the hydraulically settable sheet to fold or bend along a single line up to about 180° from horizontal without fracturing the structural material. When multiple scoring lines are made in a sheet on both sides thereof, the sheet can be bent up to 360° by being bendable in half in both directions. Before the present invention, it was not possible to fold or bend hydraulically settable materials along a single line greater than about 10°.

In general, the particular qualities of any embodiment of the present invention can be designed beforehand using a materials science and microstructural engineering approach in order to give the microstructure of the hydraulically settable structural matrix the desired properties, while at the same time remaining cognizant of the costs and other complications involved in large scale manufacturing systems. This materials science and microstructural engineering approach, instead of the traditional trial-and-error, mix-and-test approach, allows for the design of hydraulically settable materials with the desired properties of high tensile and flexural strength, low weight, low cost, and low environmental impact.

The preferred structural matrix of the sheets or containers that have the hinge of the invention integrally formed therein includes the reaction products of a cementitious or other hydraulically settable mixture. The hydraulically settable mixture will at a minimum contain a hydraulic binder, such as hydraulic cement or gypsum hemihydrate, and water. In addition, at least the location in the sheet or container where the hinge is formed will have fibers disposed therein. The inclusion of fibers allows the hydraulically settable sheets to be rolled up and later scored and folded into the desired shape of a container or other object.

In order to design the desired properties into the hydraulically settable mixture and/or the cured structural matrix, a variety of other additives can be included within the hydraulically settable mixture, such as rheology-modifying agents, dispersants, one or more aggregate materials, fibers, air entraining agents, blowing agents (often introduced during the extrusion process), or reactive metals. The identity and quantity of any additive will depend on the desired properties or performance criteria of both the hydraulically settable mixture, and the final hardened sheet.

The hydraulically settable materials utilized in the present invention can be readily recycled. Nevertheless, even if not recycled the hydraulically settable materials can be discarded and reduced to a fine granular powder which has a non-toxic composition complementary to the components of the earth into which it will be placed. This disintegration process is not dependent on biodegradation forces but will occur as a result of various natural forces which may be present, such as moisture and/or pressure.

For example, if the hydraulically settable materials are discarded into a landfill, they will crumble into a fine powder under the weight of the other garbage present. If discarded onto the ground, the forces of water and wind, and even fortuitous compressive forces such as from cars running over them or people stepping on them, will cause the hydraulically settable waste materials to be reduced to a largely inorganic, harmless granular powder in a short period of time relative to the time it usually takes for the typical disposable paper or polystyrene cup to decompose under the same circumstances.

The hydraulically settable materials utilized herein do not require the use of environmentally damaging methods or resources in order to supply the necessary raw materials. Furthermore, these materials are more environmentally neutral, do not use environmentally harmful chemicals in their manufacture, and do not create unsightly garbage which does not or very slowly degrades.

DETAILED DESCRIPTION OF THE INVENTION

The present invention is an apparatus comprising a hinge having a hydraulically settable structural matrix with a fibrous material disposed therein. The hinge of the invention is utilized in sheets and containers manufactured from hydraulically settable materials that are generally lightweight and have a high strength to bulk density ratio. The sheets and containers utilizing the hinge of the invention can be made to have a variety of densities and physical characteristics. Specific properties or qualities desired for any product can be engineered by proper selection of the material components and manufacturing processes as taught herein.

I. GENERAL DISCUSSION OF MATERIALS

A. Microstructural Engineering Design

The hydraulically settable materials used in making the sheets and containers that use the hinge of the invention were developed from the perspective of microstructural engineering and materials science in order to build into the microstructure of the material the desired properties while at the same time remaining cognizant of costs and

manufacturing complications. This microstructural engineering and materials science analysis approach, instead of the traditional trial-and-error, mix and test approach, resulted in the ability to design the hydraulically settable materials with those properties of strength, weight, malleability, insulation, cost, and environmental concerns that are necessary for rapidly manufacturing sheets and containers that use the hinge of the invention.

As a result, when the hydraulically settable mixture is molded into a sheet, it will maintain its shape (*i.e.*, support its own weight subject to minor forces, such as gravity and movement through the processing equipment) in the green state without external support. Further, from a manufacturing perspective, in order for production to be economical, it is important that the molded sheet rapidly (in a matter of minutes or even seconds) achieve sufficient strength so that it can be handled using ordinary manufacturing procedures, even though the hydraulically settable mixture may still be in a green state and not fully hardened.

Another advantage of the microstructural engineering and materials science approach utilized is the ability to develop compositions in which cross-sections of the structural matrix are more homogeneous than have been typically achieved in the prior art. Ideally, when any two given samples of about 1-2 mm^3 of the hydraulically settable matrix are taken, they will have substantially similar amounts of voids, aggregates, fibers, any other additives, and properties of the matrix.

In its simplest form, the process of using materials science in microstructurally engineering and designing a hydraulically settable material comprises characterizing, analyzing, and modifying (if necessary): (a) the aggregates, (b) the predicted particle packing, (c) the system rheology, and (d) the processing and energy of the manufacturing system.

In characterizing the aggregates, the average particle size is determined, the natural packing density of the particles (which is a function of the actual particle sizes) is determined, and the strength of the particles is ascertained. Particle packing is a primary factor for designing desired requirements of the ultimate product, such as workability, form stability, shrinkage, bulk density, insulative capabilities, tensile, compressive, and flexural strengths, elasticity, durability, and cost optimization. The particle packing is affected not only by the particle and aggregate characterization, but also by the amount of water and its relationship to the interstitial void volume of the packed aggregates.

System rheology is a function of both macro-rheology and micro-rheology. The macro-rheology is the relationship of the solid particles with respect to each other as defined by the particle packing. The micro-rheology is a function of the lubricant fraction of the system. By modification of the lubricants (which may be water, rheology-modifying agents, plasticizers, or other materials), the viscosity and yield stress can be chemically modified. The micro-rheology can also be modified physically by changing the shape and size of the particles.

Finally, the manufacturing processing can be modified to manipulate the balance between workability and form stability. As applied to the present invention, this becomes important in significantly increasing the yield stress during formation of the sheet by either chemical additive (such as by adding a rheology-modifying agent) or by adding energy to the system (such as by heating the molds). Indeed, it is this discovery of how to manipulate the hydraulically settable compositions in order to quickly increase the form stability of the compositions during the formation process that makes the present invention such a significant advancement in the art.

The microstructural engineering and materials science analysis approach used in making the hydraulically settable materials is discussed in more detail in the current co-pending patent application Serial No. 08/101,500, entitled "Methods and Apparatus for Manufacturing Moldable Hydraulically Settable Sheets Used in Making Containers, Printed Materials, and Other Objects," filed August 3, 1993 in the names of Per Just Andersen, Ph.D. and Simon K. Hodson, which is incorporated herein by specific reference.

B. Hydraulically Settable Materials

The materials used to manufacture the hydraulically settable sheets and containers that use the hinge of the present invention develop strength through the chemical reaction of water and a hydraulic binder, such as hydraulic cement, calcium sulfate (or gypsum) hemihydrate, and other substances that harden after being exposed to water. The term "hydraulically settable materials" as used in this specification and the appended claims includes any material with a structural matrix and strength properties that are derived from a hardening or curing of a hydraulic binder. These include cementitious materials, plasters, and other hydraulically settable materials as defined herein. The hydraulically settable binders used in the present invention are to be distinguished from other cements or binders such as polymerizable, water insoluble organic cements, glues, or adhesives.

The terms "hydraulically settable materials," "hydraulic cement materials," or "cementitious materials," as used herein, are intended to broadly define compositions and materials that contain both a hydraulically settable binder and water, regardless of the extent of hydration or curing that has taken place. Hence, it is intended that the term "hydraulically settable materials" shall include hydraulic paste or hydraulically settable mixtures in a green (*i.e.*, unhardened) state, as well as hardened hydraulically settable or concrete products.

1. Hydraulically Settable Binders

The terms "hydraulically settable binder" or "hydraulic binder" as used in this specification and the appended claims are intended to include any inorganic binder such as hydraulic cement, gypsum hemihydrate, or calcium oxide which develop strength properties and hardness by chemically reacting with water and, in some cases, with carbon dioxide in the air and water. The terms "hydraulic cement" or "cement" as used in this specification and the appended claims are intended to include clinker and crushed, ground, milled, and processed clinker in various stages of pulverization and in various particle sizes.

Examples of typical hydraulic cements that can be utilized include: the broad family of portland cements (including ordinary portland cement without gypsum), MDF cement, DSP cement, Densit-type cements, Pyrament-type cements, calcium aluminate cements (including calcium aluminate cements without set regulators), plasters, silicate cements (including ß-dicalcium silicates, tricalcium silicates, and mixtures thereof), gypsum cements, phosphate cements, high alumina cements, microfine cements, slag cements, magnesium oxychloride cements, and aggregates coated with microfine cement particles.

The term "hydraulic cement" is also intended to include other cements known in the art, such as α-dicalcium silicate, which can be made hydraulic under hydrating conditions within the scope of the present invention. The basic chemical components of, *e.g.*, portland cement include CaO, SiO_2, Al_2O_3, Fe_2O_3, MgO, and SO_3, in various combinations and proportions. These react together in the presence of water in a series of complex reactions to form insoluble calcium silicate hydrates, carbonates (from CO_2 in the air and added water), sulfates, and other salts or products of calcium and magnesium, together with hydrates thereof. The aluminum and iron constituents are thought to be incorporated into elaborate complexes within the above mentioned insoluble salts. The cured cement product is a complex matrix of insoluble hydrates and salts that are

complexed and linked together much like stone. This material is highly inert and has both physical and chemical properties similar to those of natural stone or dirt.

Gypsum is also a hydraulically settable binder that can be hydrated to form a hardened binding agent. One hydratable form of gypsum is calcium sulfate hemihydrate, commonly known as "gypsum hemihydrate." The hydrated form of gypsum is calcium sulfate dihydrate, commonly known as "gypsum dihydrate." Calcium sulfate hemihydrate can also be mixed with calcium sulfate anhydride, commonly known as "gypsum anhydrite" or simply "anhydrite."

Although gypsum binders or other hydraulic binders such as calcium oxide are generally not as strong as hydraulic cement, high strength may not be as important as other characteristics (*e.g.*, the rate of hardening) in some applications. In terms of cost, gypsum and calcium oxide have an advantage over hydraulic cement because they are somewhat less expensive. Moreover, in the case where the hydraulically settable material contains a relatively high percentage of weak, lighter weight aggregates (such as perlite), the aggregates will often comprise a "weak link" within the structural matrix. At some point, adding a stronger binder may be inefficient because the binder no longer contributes its higher potential strength due to a high content of weaker aggregates.

In addition, gypsum hemihydrate is known to set up or harden in a much shorter time period than traditional cements. In fact, in use with the present invention, it will harden and attain most of its ultimate strength within about thirty minutes. Hence, gypsum hemihydrate can be used alone or in combination with other hydraulically settable materials within the scope of the present invention.

Terms such as "hydrated" or "cured" hydraulically settable mixture, material, or matrix refer to a level of substantial water-catalyzed reaction which is sufficient to produce a hydraulically settable product having a substantial amount of its potential or final maximum strength. Nevertheless, hydraulically settable materials may continue to hydrate long after they have attained significant hardness and a substantial amount of their final maximum strength.

Terms such as "green" or "green state" are used in conjunction with hydraulically settable mixtures that have not achieved a substantial amount of their final strength, regardless of whether such strength is derived from artificial drying, curing, or other means. Hydraulically settable mixtures are said to be "green" or in a "green state" just prior and subsequent to being molded into the desired shape. The moment when a hydraulically settable mixture is no longer "green" or in a "green state" is not necessarily a clear-cut line of demarcation, since such mixtures generally attain a substantial amount

of their total strength only gradually over time. Hydraulically settable mixtures can, of course, show an increase in "green strength" and yet still be "green." For this reason, the discussion herein often refers to the form stability of the hydraulically settable material in the green state.

Preferable hydraulic binders include white cement, portland cement, microfine cement, high alumina cement, slag cement, gypsum hemihydrate, and calcium oxide, mainly because of their low cost and suitability for the manufacturing processes used in the present invention. This list of cements is by no means exhaustive, nor in any way is it intended to limit the types of binders which would be useful in making the hydraulically settable containers that use the hinge of the invention.

The present invention may include other types of cementitious compositions such as those discussed in copending patent application Serial No. 07/981,615, filed November 25, 1992 in the names of Hamlin M. Jennings, Ph.D. and Simon K. Hodson and entitled "Methods of Manufacture And Use For Hydraulically Bonded Cement," which is a continuation-in-part of patent application Serial No. 07/856,257, filed March 25, 1992 in the names of Hamlin M. Jennings, Ph.D. and Simon K. Hodson, and entitled "Hydraulically Bonded Cement Compositions and Their Methods of Manufacture and Use" (now abandoned). In these applications, powdered hydraulic cement is placed in a near net final position and compacted prior to the addition of water for hydration. For purposes of disclosing the use of such compositions, the forgoing patent applications are incorporated herein by specific reference.

Additional types of hydraulic cement compositions include those wherein carbon dioxide is mixed with hydraulic cement and water. Hydraulic cement compositions made by this method are known for their ability to more rapidly achieve green strength. This type of hydraulic cement composition is discussed in copending patent application Serial No. 07/418.027 filed October 10, 1989 in the names of Hamlin M. Jennings, Ph.D. and Simon K. Hodson and entitled "Process for Producing Improved Building Material and Products Thereof," wherein water and hydraulic cement are mixed in the presence of a carbonate source selected from the group consisting of carbon dioxide, carbon monoxide, carbonate salts. and mixtures thereof. For purposes of disclosure, the forgoing patent application is incorporated herein by specific reference.

In many situations, it may not be desirable for the sheet (or a container made therefrom) to be water soluble. Unfortunately, certain materials which might be desirable to incorporate into such containers dissolve in water. An important advantage of using a hydraulically settable mixture is that the resulting structural matrix is generally water

insoluble (at least over the period of time during which use of the product is intended), which allows it to encapsulate the water soluble aggregates or other materials added to the hydraulically settable mixture. Hence, an otherwise water soluble component can be incorporated into the greatly insoluble hydraulically settable matrix and impart its advantageous properties and characteristics to the final product.

Nevertheless, in order to design a disposable sheet or container product which will more readily decompose or disintegrate after it has fulfilled its intended use, it may be desirable for the sheet or container to break down in the presence of water or moisture. One of the advantages of the microstructural engineering and materials science approach as applied to the materials of the present invention is the ability to design into the hydraulically settable structural matrix the desired properties of water resistance or solubility. In order to obtain a sheet or container that readily decomposes in the presence of water, it will generally be necessary to decrease the amount of hydraulic binder within the material. Hence, the degree of water solubility or insolubility is generally related to the concentration of hydraulic binder, particularly hydraulic cement, within the hydraulically settable mixture. In most cases, adding more hydraulic binder will make the container less soluble in water.

2. <u>Hydraulic Paste</u>

In each embodiment of the present invention, the hydraulic paste or cement paste is the key constituent which eventually gives the sheet or container the ability to set up and develop strength properties. The term "hydraulic paste" refers to a hydraulic binder mixed with water. More specifically, the term "cement paste" refers to hydraulic binder such as a cement mixed with water. The terms "hydraulically settable," "hydraulic," or "cementitious" mixture refer to a hydraulic binder such as a cement paste to which aggregates, fibers, rheology-modifying agents, dispersants, or other materials have been added, whether in the green state or after it has hardened and/or cured. The other ingredients added to the hydraulic paste serve the purpose of altering the properties of the unhardened, as well as the final hardened product, including, but not limited to, strength, shrinkage, flexibility, bulk density, insulating ability, color, porosity, surface finish, and texture.

Although the hydraulic binder is understood as the component which allows the hydraulically settable mixture to set up, to harden, and to achieve much of the strength properties of the material, certain hydraulic binders also aid in the development of better early cohesion and green strength. For example, hydraulic cement particles undergo

early gelating reactions with water even before becoming hard, which can contribute to the internal cohesion of the mixture.

It is believed that aluminates, such as those more prevalent in portland grey cement (in the form of tricalcium aluminates) are responsible for a colloidal interaction between the cement particles during the earlier stages of hydration. This in turn causes a level of flocculation/gelation to occur between the cement particles. The gelating, colloidal, and flocculating affects of such binders increases the moldability (*i.e.*, plasticity) of hydraulically settable mixtures made therefrom.

As set forth more fully below, additives such as fibers and rheology-modifying agents can make substantial contributions to the hydraulically settable materials in terms of tensile, flexural, and compressive strengths. Nevertheless, even where high concentrations of fibers and/or rheology-modifying agents are included and contribute substantially to the tensile and flexural strengths of the hardened material, the hydraulic binder continues to add substantial amounts of compressive strength to the final hardened material. In the case of hydraulic cement, the binder also substantially reduces the solubility of the hardened material in water.

The percentage of the hydraulically settable binder within the overall mixture varies depending on the identity of the other added constituents. The hydraulically settable binder is preferably added in an amount ranging from between about 5% to about 95% by weight of the wet hydraulically settable mixture. From the disclosure and examples set forth herein, it will be understood that this wide range of weight percent covers hydraulically settable mixtures used to manufacture foam-like or clay-like sheets and containers.

It will be appreciated from the foregoing that embodiments within the scope of the present invention will vary from a very lightweight "foam-like" product to a somewhat higher density "clay-like" product. Either foam-like or clay-like materials can readily be molded into sheet products that can be handled much like paper, cardboard, plastic, or even a sheet of metal. Within these broader categories will be other variations and differences that will require varying quantities and identities of the components. The components and their relative quantities may substantially vary depending upon the specific container or other product to be made.

Generally, when making a "foam-like" sheet product, it will be preferable to include the hydraulic binder within the range from between about 1% to about 70% by volume of the total solids of the hydraulically settable mixture, and more preferably within the range from between about 5% to about 30% by volume. When making a

"clay-like" sheet product, it will be preferable to include the hydraulic binder within the range from between 1% to about 70% by volume of the total solids of the hydraulically settable mixture, preferably within the range from about 5% to about 30% by volume, and most preferably within a range from about 5% to about 15% by volume.

Despite the foregoing, it will be appreciated that all concentrations and amounts are critically dependent upon the qualities and characteristics that are desired in the final product. For example, in a very thin wall structure (even as thin as 0.05 mm) where strength is needed, it may be more economical to have a very high percentage of hydraulic binder with little or no aggregate. In such a case, it may be desirable to include a high amount of fiber to impart flexibility and toughness. Conversely, in a product in which high amounts of air are incorporated, there may be a greater percentage of the rheology-modifying agent, a smaller amount of cement, and larger amounts of lightweight aggregates. Such materials can literally be as light as lightweight polystyrene foam products.

The other important constituent of hydraulic paste is water. By definition, water is an essential component of the hydraulically settable materials within the scope of the present invention. The hydration reaction between the hydraulic binder and water yields reaction products which give the hydraulically settable materials the ability to set up and develop strength properties.

In most applications of the present invention, it is important that the water to cement ratio be carefully controlled in order to obtain a hydraulically settable mixture which after molding, extrusion, and/or calendering is self-supporting in the green state. Nevertheless, the amount of water to be used is dependent upon a variety of factors, including the types and amounts of hydraulic binder, aggregates, fibrous materials, rheology-modifying agents, and other materials or additives within the hydraulically settable mixture, as well as the molding or forming process to be used, the specific product to be made, and its properties.

The preferred amount of added water within any given application is primarily dependent upon two key variables: (1) the amount of water which is required to react with and hydrate the binder; and (2) the amount of water required to give the hydraulically settable mixture the necessary rheological properties and workability.

In order for the green hydraulically settable mixture to have adequate workability, water must generally be included in quantities sufficient to wet each of the particular components and also to at least partially fill the interstices or voids between the particles (including, *e.g.*, binder particles, aggregates, and fibrous materials). If water soluble

additives are included, enough water must be added to dissolve or otherwise react with the additive. In some cases, such as where a dispersant is added, workability can be increased while using less water.

The amount of water must be carefully balanced so that the hydraulically settable mixture is sufficiently workable, while at the same time recognizing that lowering the water content increases both the green strength and the final strength of the hardened product. Of course, if less water is initially included within the mixture, less water must be removed in order to allow the product to harden.

The appropriate rheology to meet these needs can be defined in terms of yield stress. The yield stress of the hydraulically settable mixture will usually be in the range from between about 5 kPa to about 5,000 kPa, with the more preferred mixtures having a yield stress within a range from about 100 kPa to about 1,000 kPa, and the most preferred mixtures having a yield stress in the range from about 200 kPa to about 700 kPa. The desired level of yield stress can be (and may necessarily have to be) adjusted depending on the particular molding process being used to form the food or beverage container.

In each of the molding processes, it may be desirable to initially include a relatively high water to cement ratio during or shortly after the molding process. One of the important features of the present invention as compared to the manufacture of paper is that the amount of water in the initial mixture is much less; hence, the yield stress is greater for the hydraulically settable mixtures. The result is that the total amount of water that must be removed from the initial mixture to obtain a self-supporting material (*i.e.*, a form stable material) is much less in the case of the present invention when compared to the manufacture of paper.

Nevertheless, one skilled in the art will understand that when more aggregates or other water absorbing additives are included, a higher water to hydraulically settable binder ratio is necessary in order to provide the same level of workability and available water to hydrate the hydraulically settable binder. This is because a greater aggregate concentration provides a greater volume of interparticulate interstices or voids which must be filled by the water. Porous, lightweight aggregates can also internally absorb significant amounts of water due to their high void content.

Both of the competing goals of greater workability and high green strength can be accommodated by initially adding a relatively large amount of water and then driving off much of the water as steam during the molding process, usually by the use of heated rollers or drying tunnels.

Based on the foregoing qualifications, typically hydraulically settable mixtures within the scope of the present invention will have a water to hydraulically settable binder ratio within a range from about 0.1:1 to about 10:1, preferably about 0.3:1 to about 4:1, and most preferably from about 1:1 to about 3:1. The total amount of water remaining after drying the material to remove excess water will range up to about 20% by volume with respect to the dry, hardened hydraulically settable sheet or container.

It should be understood that the hydraulic binder has an internal drying effect on the hydraulically settable mixture because binder particles chemically react with water and reduce the amount of free water within the interparticulate interstices. This internal drying effect can be enhanced by including faster reacting hydraulic binders such as gypsum hemihydrate along with slower reacting hydraulic cement.

According to a preferred embodiment of the present invention, it has been found desirable that the hydraulic binder and water be mixed in a high shear mixer such as that disclosed and claimed in U.S. Patent No. 4,225,247 entitled "Mixing and Agitating Device"; U.S. Patent No. 4,552,463 entitled "Method and Apparatus for Producing a Colloidal Mixture"; U.S. Patent No. 4,889,428 entitled "Rotary Mill"; U.S. Patent No. 4,944,595 entitled "Apparatus for Producing Cement Building Materials"; and U.S. Patent No. 5,061,319 entitled "Process for Producing Cement Building Material." For purposes of disclosure, the forgoing patents are incorporated herein by specific reference. High shear mixers within the scope of these patents are available from E. Khashoggi Industries of Santa Barbara, California, the assignee of the present invention.

The use of a high shear mixer results in a more homogeneous hydraulically settable mixture, which results in a product with higher strength. Furthermore, high shear mixers can be utilized to entrain significant amounts of air into the hydraulically settable mixture to create "foam-like" products.

C. Fibers.

As used in the specification and appended claims, the terms "fibers" and "fibrous materials" include both inorganic fibers and organic fibers. Fibers are a particular kind of aggregate that may be added to the hydraulically settable mixture to increase the cohesion, elongation ability, deflection ability, toughness, fracture energy, and flexural, tensile, and, on occasion, even compressive strengths. Fibrous materials reduce the likelihood that the hydraulically settable sheet will shatter when a strong cross-sectional force is applied.

In evaluating potential fibers for use in the container and hinge material, important characteristics to consider are: the physical properties of the fibers (e.g., length and diameter, tensile strength, and wetability/dispersability), cost, reliability of supply (quantity and consistency), the relative level of contaminants in the fiber (e.g.,lignin, pectin, fats/waxes, etc.) and the acceptability of the fiber to food contact applications. The use of fibers dramatically increases the fracture energy of the cementitious materials, which make the resulting containers and hinges particularly useful for packaging, storing, and shipping goods.

Examples of fibers that may be utilized singly or in a variety of mixtures include glass fibers, silica fibers, ceramic fibers (such as alumina, silica nitride, silica carbide, graphite), rock wool, metal fibers, carbon fibers, and synthetic polymer fibers such as polypropylene, polyethylene, nylon, or rayon fibers. Fibers extracted from plant leaves and stems may be used, as well as any naturally occurring fiber comprised of cellulose. Such fibers are available from wood and paper pulp (e.g., wood flour or saw dust), wood fibers (both hardwood or softwood such as southern pine), recycled paper, cotton, cotton linters, abaca (Manila hemp), sisal, jute, sunn hemp, flax, and bagasse. Recycled paper fibers are somewhat less desirable because of the fiber degradation that occurs during the original paper manufacturing process, as well as in the recycling process. Any equivalent fiber, however, which imparts strength and flexibility is also within the scope of the present invention.

Preferred fibers include glass fibers, cellulose fibers (from unbleached kraft pulp), abaca fibers (extracted from a Philippine hemp plant related to the banana), bagasse, wood fibers, ceramic fibers, and cotton. When glass fibers are used they are preferably pretreated to be alkali resistant. Glass fibers such as Cemfill® are available from Pilkington Corp. in England. Abaca fibers are available from Isarog Inc. in the Philippines.

Fibers or other aggregates may also be formed in the hydraulically settable sheets as inorganic precipitates in situ. Such precipitates can be in the form of polymerized silicates, alumino-silicate gels, etc.

The fibers used to make the hydraulically settable sheets used in the present invention preferably have a high length to width ratio (or "aspect ratio") because longer, narrower fibers can impart more strength to the matrix without significantly adding bulk and mass to the mixture. The fibers should have an aspect ratio of at least about 10:1, preferably at least about 100:1. An aspect ratio of between about 200:1 to 300:1 is most preferred.

Preferred fibers should also have a length that is several times the diameter of the hydraulically settable binder particles. Fibers having a length that is at least about twice the average diameter of the hydraulic binder particles will work, while fibers having a length at least about 10 times the average diameter of the hydraulic binder particles is preferred, with at least about 100 times being more preferred, and even about 1000 times being very useful. High fiber length to binder particle ratios can be achieved by either increasing the absolute length of the added fibers or, alternatively, by using a more finely milled binder.

The amount of fibers added to the hydraulically settable matrix will vary depending upon the desired properties of the final product, with strength, toughness, flexibility, and cost being the principal criteria for determining the amount of fiber to be added in any mix design. In most cases, fibers will be added in an amount within the range from about 0.2% to about 50% by volume of the total solids of the hydraulically settable mixture, more preferably within the range from about 1% to about 30% by volume, and most preferably within the range from about 5% to about 15% by volume.

It will be appreciated, however, that the strength of the fiber is a very important feature in determining the amount of the fiber to be used. The stronger the tensile strength of the fiber, the less the amount that must be used to obtain the same level of tensile strength in the resulting product. Of course, while some fibers have a high tensile strength, other types of fibers with a lower tensile strength may be more elastic. Hence, a combination of two or more fibers may be desirable in order to obtain a resulting product that maximizes multiple characteristics, such as high tensile strength and high elasticity.

In addition, while ceramic fibers are generally far more expensive than naturally occurring or glass fibers, their use will nevertheless be economical in some cases due to their far superior tensile strength properties. Obviously the use of more expensive fibers becomes more economical as the cost restraints of hydraulically settable sheets are relaxed, such as where a comparable sheet made from competing materials is relatively expensive.

It should be understood that the fibers used within the scope of the present invention differ from fibers typically employed in making paper or cardboard sheets, primarily in the way in which the fibers are processed. In the manufacture of paper, either a Kraft or a sulphite process is typically used to form the pulp sheet. In the Kraft process, the pulp fibers are "cooked" in a NaOH process to break up the fibers. In a sulphite process, acid is used in the fiber disintegration process.

In both of these processes, the fibers are first processed in order to release lignins locked within the fiber walls. However, in order to release the lignins from the fiber, some of the strength of the fiber is lost. Because the sulfite process is even more severe, the strength of the paper made by a sulphite process will generally have only about 70% of the strength of paper made by the Kraft process. (Hence, to the extent wood fibers are included, those processes using a Kraft process are preferred.)

It should also be understood that some fibers, such as southern pine and abaca, have high tear and burst strengths, while others, such as cotton, have lower strength but greater flexibility. In the case where both flexibility and high tear and burst strength is desired, a mixture of fibers having the various properties can be added to the mixture.

Fibers are particularly important where the sheet has been scored and is expected to bend over a larger angle. In addition, the properties imparted to the hardened sheets by the fibers can be increased by unidirectionally or bidirectionally orienting the fibers within the hydraulically settable rolled sheet. Depending on the shape of the extruder die head, the extrusion process itself will tend to orient the fibers in the "Y" (or longitudinal) direction. The sheet thickness reduction process, during which the sheet is also elongated, further orients the fibers in the "Y" direction.

In addition, by using a pair of rollers having different orientations in the "Z" direction (or normal to the surface of the sheet), such as by using a flat roller paired with a conical roller, a percentage of the fibers can be oriented in the "X" (or width-wise) direction. This is thought to occur because the conical roller can widen the sheet in the "X" direction. In this way a sheet having bidirectionally oriented fibers can be manufactured. As a result, the desired strength characteristics can be engineered into the resultant sheet.

D. <u>Rheology-modifying Agents</u>.

The inclusion of a rheology-modifying agent acts to increase the plastic or cohesive nature of the hydraulically settable mixture so that it behaves more like a moldable clay. The rheology-modifying agent tends to thicken the hydraulically settable mixture by increasing the yield stress of the mixture without greatly increasing the viscosity of the mixture. Raising the yield stress in relation to the viscosity makes the material more plastic-like and moldable, while greatly increasing the subsequent form stability or green strength.

A variety of natural and synthetic organic rheology-modifying agents may be used that have a wide range of properties, including viscosity and solubility in water. For

example, where it is desirable for the sheet or container to more quickly break down into environmentally benign components, it may be preferable to use a rheology-modifying agent that is more water soluble. Conversely, in order to design a material capable of withstanding prolonged exposure to water, it may be preferable to use a rheology-modifying agent that is less soluble in water or to use a high content of the hydraulic binder with respect to the rheology-modifying agent.

The various rheology-modifying agents contemplated by the present invention can be roughly organized into the following categories: (1) polysaccharides and derivatives thereof, (2) proteins and derivatives thereof, and (3) synthetic organic materials. Polysaccharide rheology-modifying agents can be further subdivided into (a) cellulose-based materials and derivatives thereof, (b) starch based materials and derivatives thereof, and (c) other polysaccharides.

Suitable cellulose-based rheology-modifying agents include, for example, methylhydroxyethylcellulose, hydroxymethylethylcellulose, carboxymethylcellulose, methylcellulose, ethylcellulose, hydroxyethylcellulose, hydroxyethylpropylcellulose, etc. The entire range of possible permutations is enormous and cannot be listed here, but other cellulose materials that have the same or similar properties as these would also work well.

Suitable starch based materials include, for example, amylopectin, amylose, seagel, starch acetates, starch hydroxyethyl ethers, ionic starches, long-chain alkylstarches, dextrins, amine starches, phosphate starches, dialdehyde starches, etc. Other natural polysaccharide based rheology-modifying agents include, for example, alginic acid, phycocolloids, agar, gum arabic, guar gum, locust bean gum, gum karaya, and gum tragacanth.

Suitable protein-based rheology-modifying agents include, for example, Zein® (a prolamine derived from corn), collagen derivatives extracted from animal connective tissue such as gelatin and glue, and casein (the principal protein in cow's milk). Suitable synthetic organic plasticizers include, for example, polyvinyl pyrrolidone, polyethylene glycol, polyvinyl alcohol, polyvinylmethyl ether, polyacrylic acids, polyacrylic acid salts, polyvinylacrylic acids, polyvinylacrylic acid salts, polyacrylimides, ethylene oxide polymers, polylactic acid, synthetic clay, and latex (which is a styrene-butadiene copolymer). The rheology of polylactic acid is significantly modified by heat and could be used alone or in combination with any other of the foregoing rheology-modifying agents.

A currently preferred rheology-modifying agent is methylhydroxyethylcellulose, examples of which are Tylose® FL 15002 and Tylose® 4000, both of which are available from Hoechst Aktiengesellschaft of Frankfurt, Germany. Lower molecular weight rheology-modifying agents such as Tylose® 4000 can act to plasticize the mixture rather than thicken it, which helps during extrusion or rolling procedures.

More particularly, lower molecular weight rheology-modifying agents improve the internal flow of the hydraulically settable mixture during molding processes by lubricating the particles. This reduces the friction between the particles as well as between the mixture and the adjacent mold surfaces. Although a methylhydroxyethylcellulose rheology-modifying agent is preferred, almost any non-toxic rheology-modifying agent (including any listed above) which imparts the desired properties would be appropriate.

Another preferred rheology-modifying agent that can be used instead of, or in conjunction with, Tylose® is polyethylene glycol having a molecular weight of between about 20,000 and 35,000. Polyethylene glycol works more as a lubricant and adds a smoother consistency to the mixture. For this reason, polyethylene glycol might be referred to more precisely as a "plasticizer." In addition, it gives the molded hydraulically settable material a smoother surface. Finally, polyethylene glycol can create a coating around soluble components of the mixture and thereby render the hardened product less water soluble.

Starch-based rheology-modifying agents are of particular interest within the scope of the present invention because of their comparatively low cost compared to cellulose-based rheology-modifying agents such as Tylose®. Although starches typically require heat and/or pressure in order to gelate, starches may by modified and prereacted so that they can gel at room temperature. The fact that starches, as well as many of the other rheology-modifying agents listed above, have a variety of solubilities, viscosities, and rheologies allows for the careful tailoring of the desired properties of a mix design so that it will conform to the particular manufacturing and performance criteria of a particular food or beverage container.

It is also within the scope of this invention to include rheology-modifying agents by utilizing various mixtures of cellulose-based materials, protein-based materials, starch-based materials, and organic plasticizers.

The rheology-modifying agent that can be used in the hydraulically settable materials can be included in an amount within the range from about 0.1% to about 30% by volume of the total solids of the hydraulically settable mixture, preferably within the

range from about 0.5% to about 15% by volume, and most preferably within the range from about 1% to about 10% by volume of the hydraulically settable mixture.

E. **Dispersants.**

The term "dispersant" is used hereinafter to refer to the class of materials that can be added to reduce the viscosity and yield stress of the hydraulically settable mixture. A more detailed description of the use of dispersants may be found in the Master's Thesis of Andersen, P.J., "Effects of Organic Superplasticizing Admixtures and Their Components on Zeta Potential and Related Properties of Cement Materials" (1987). For purposes of disclosure, the above-referenced article is incorporated herein by specific reference.

Dispersants generally work by being adsorbed onto the surface of the hydraulic binder particles and/or into the near colloid double layer of the binder particles. This creates a negative charge around the surfaces of particles, causing them to repel each other. This repulsion of the particles adds "lubrication" by reducing the "friction" or attractive forces that would otherwise cause the particles to have greater interaction. Because of this, less water can be added initially while maintaining the workability of the hydraulically settable mixture.

Greatly reducing the viscosity and yield stress may be desirable where clay-like properties, cohesiveness, and/or form stability are less important. Adding a dispersant aids in keeping the hydraulically settable mixture workable even when very little water is added, particularly where there is a "deficiency" of water. Hence, adding a dispersant allows for an even greater deficiency of water, although the molded sheet may have somewhat less form stability if too much dispersant is used. Nevertheless, including less water initially will theoretically yield a stronger final cured sheet according to the Feret Equation.

Whether or not there is a deficiency of water is both a function of the stoichiometric amount of water required to hydrate the binder and the amount of water needed to occupy the interstices between the particles in the hydraulically settable mixture, including the hydraulic binder particles themselves and the particles within the aggregate material and/or fibrous material. As stated above, improved particle packing reduces the volume of the interstices between the hydraulic binder and aggregate particles and, hence, the amount of water necessary to fully hydrate the binder and maintain the workability of the hydraulically settable mixture by filling the interstitial space.

However, due to the nature of the coating mechanism of the dispersant, the order in which the dispersant is added to the mixture is often critical. If a flocculating/gelating agent such as Tylose® is added, the dispersant must be added first and the flocculating agent second. Otherwise, the dispersant will not be able to become adsorbed on the surface of the hydraulic binder particles as the Tylose® will be irreversibly adsorbed onto the surface of the particles, thereby bridging them together rather than causing them to repel each other.

A preferred dispersant is sulfonated naphthalene-formaldehyde condensate, an example of which is WRDA 19, which is available from W.R. Grace, Inc. located in Baltimore, Maryland. Other dispersants which would work well include sulfonated melamine-formaldehyde condensate, lignosulfonate, and acrylic acid.

The amount of added dispersant will generally range up to about 3% by volume of the total solids of the hydraulically settable mixture, more preferably within the range of between about 0.1% to about 2% by volume, and most preferably within the range of between about 0.2% to about 1% by volume. However, it is important not to include too much dispersant as it tends to retard the hydration reactions between, *e.g.*, hydraulic cement and water. Adding too much dispersant can, in fact, prevent hydration, thereby destroying the binding ability of the cement paste altogether.

The dispersants contemplated within the present invention have sometimes been referred to in the concrete industry as "superplasticizers." In order to better distinguish dispersants from rheology-modifying agents, which often act as plasticizers, the term "superplasticizer" will not be used in this specification.

F. <u>Nonfibrous Aggregates</u>.

Aggregates common in the concrete industry may be employed in the hydraulically settable mixtures used in the present invention. Such aggregates, however, often must be more finely ground due to the size limitations imposed by the generally thin-walled structures that utilize the hinge of the invention. The diameter of the aggregates used will most often be less than about 25% of the smallest cross-section of the structural matrix of the sheet.

Aggregates may be added to increase the strength, decrease the cost by acting as a filler, decrease the weight, and/or increase the insulation ability of the resultant hydraulically settable materials. Aggregates are also useful for creating a smooth surface finish, particularly plate-like aggregates. Examples of useful aggregates include perlite, vermiculite, sand, gravel, rock, limestone, sandstone, glass beads, aerogels, xerogels,

seagel, mica, clay, synthetic clay, alumina, silica, fly ash, silica fume, tabular alumina, kaolin, micro spheres, hollow glass spheres, porous ceramic spheres, gypsum dihydrate, calcium carbonate, calcium aluminate, cork, seeds, lightweight polymers, xonotlite (a crystalline calcium silicate gel), lightweight expanded clays, unreacted cement particles, pumice, exfoliated rock and other geologic materials. Unreacted cement particles may also be considered to be "aggregates" in the broadest sense of the term. Even discarded hydraulically settable materials, such as discarded sheets or containers can be employed as aggregate fillers and strengtheners.

The amount of the aggregate will vary depending upon the particular application or purpose, from no added aggregate up to about 90% by volume of the total solids of the hydraulically settable mixture, more preferably within the range from between about 5% to about 70% by volume, and most preferably from between about 20% to about 50% by volume of the mixture.

Both clay and gypsum are particularly important aggregate materials because of their ready availability, extreme low cost, workability, ease of formation, and because they can also provide a degree of binding and strength if added in high enough amounts. Clay is a general term used to identify all earths that form a paste with water and harden when dried. The predominant clays include silica and alumina (used for making pottery, tiles, brick, and pipes) and kaolinite. The two kaolinitic clays are anauxite, which has the chemical formula $Al_2O_3 \cdot 3SiO_2 \cdot 2H_2O$, and montmorillonite, which has the chemical formula $Al_2O_3 \cdot 4SiO_2 \cdot H_2O$. Clays may also contain a wide variety of other substances such as iron oxide, titanium oxide, calcium oxide, zirconium oxide, and pyrite.

In addition, although clays can obtain hardness even without being fired, such unfired clays are vulnerable to water degradation and exposure, are extremely brittle, and have low strength. Nevertheless, clay makes a good, inexpensive aggregate within the hydraulically settable structural matrix. In fact, clay may even contribute a degree of binding to the hydraulically settable mixture if included in large enough amounts.

Similarly, gypsum hemihydrate is also hydratable and forms the dihydrate of calcium sulfate in the presence of water. Thus, gypsum may exhibit the characteristics of both an aggregate and a binder depending on whether (and the concentration of) the hemihydrate or dihydrate form is added to a hydraulically settable mixture.

It is often preferable according to the present invention to include a plurality of differently sized and graded aggregates capable of more completely filling the interstices between the aggregate and hydraulic binder particles. Optimizing the particle packing density reduces the amount of water necessary to obtain adequate workability by

eliminating spaces which would otherwise be filled with interstitial water, often referred to as "capillary water." In addition, using less water increases the strength of the final hardened product (according to the Feret Equation).

In order to optimize the packing density, differently sized aggregates with particle sizes ranging from as small as about 0.5 microns to as large as about 2 mm may be used. The desired purpose and thickness of the resulting product will dictate the appropriate particle sizes of the various aggregates to be used.

In certain preferred embodiments of the present invention, it may be desirable to maximize the amount of the aggregates within the hydraulically settable mixture in order to maximize the properties and characteristics of the aggregates (such as qualities of strength, low density, or high insulation). The use of particle packing techniques may be employed within the hydraulically settable material in order to maximize the amount of the aggregates.

A detailed discussion of particle packing can be found in the following article coauthored by one of the inventors of the present invention: Johansen, V. & Andersen, P.J., "Particle Packing and Concrete Properties," Materials Science of Concrete II at 111-147, The American Ceramic Society (1991). Further information is available in the Doctoral Dissertation of Andersen, P.J. "Control and Monitoring of Concrete Production —— A Study of Particle Packing and Rheology," The Danish Academy of Technical Sciences. For purposes of teaching particle packing techniques, the disclosures of the foregoing article and thesis are incorporated herein by specific reference.

In embodiments in which it is desirable to obtain a food or beverage container with high insulation capability, it may be preferable to incorporate into the hydraulically settable matrix a lightweight aggregate which has a low thermal conductivity, or "k-factor" (defined as W/m·K), which is roughly the reciprocal of the expression commonly used in the United States for thermal resistance, or "R-factor," which is generally defined as having units of hr-ft^2°F/BTU. The term "R-factor" is most commonly used in the United States to describe the overall thermal resistance of a given material without regard to the thickness of the material. However, for purposes of comparison, it is common to normalize the R-factor to describe thermal resistance per inch of thickness of the material in question, or hr-ft^2°F/BTU-in.

For purposes of this application, the insulation ability of a given material will hereinafter be expressed only in terms of the IUPAC method of describing thermal conductivity, or k-factor. The conversion of thermal resistance expressed in British units (hr-ft^2°F/BTU-in) to IUPAC units can be performed by multiplying the normalized R-

factor by 6.9335 and then taking the reciprocal of the product. Generally, aggregates having a very low k-factor also contain large amounts of trapped interstitial space or air, which also tends to greatly reduce the strength of such aggregates. Therefore, concerns for insulation and strength tend to compete and should be carefully balanced when designing a particular mix design.

G. Air Voids.

Where insulation, not strength, is the overriding factor, it may be desirable to incorporate tiny air voids within the hydraulically settable structural matrix. Air voids can be introduced by adding an air entraining agent and mixing the hydraulically settable mixture in a high speed mixer, such as those discussed above.

Suitable air entrainment agents include commonly used surfactants. One currently preferred surfactant is a polypeptide alkylene polyol (Mearlcrete® Foam Liquid).

In conjunction with the surfactant, it will be necessary to stabilize the entrained air within the material using a stabilizing agent like Mearlcel 3532®, a synthetic liquid anionic biodegradable solution. Both of the Mearlcrete® and Mearlcel® products are available from the Mearl Corporation in New Jersey. Another foaming and air-entraining agent is vinsol resin. In addition, the rheology-modifying agent can act to stabilize the entrained air. Different air-entraining agents and stabilizing agents impart different degrees of foam stability to the hydraulically settable mixture, and they should be chosen in order to impart the properties that are best suited for a particular manufacturing process.

During the entrainment of air, the atmosphere above the high speed mixer can be saturated with a gas such as carbon dioxide, which has been found to cause an early false setting and create form and foam stability of the hydraulically settable mixture. The early false setting and foam stability is thought to result from the reaction of CO_2 and hydroxide ions within the hydraulically settable mixture to form carbonate ions, which in turn can form insoluble precipitates with calcium and other cations within the mixture.

Foam stability helps maintain the dispersion and prevents the agglomeration of the air voids within the uncured hydraulically settable mixture. Failure to prevent the coalescence of the air voids actually decreases the insulation effect, while greatly decreasing the strength, of the cured hydraulically settable mixture. Raising the pH, increasing the concentration of soluble alkali metals such as sodium or potassium, adding a stabilizing agent such as a polysaccharide rheology-modifying agent, and carefully

adjusting the concentrations of surfactant and water within the hydraulically settable mixture all help to increase the foam stability of the mixture.

Air voids may alternatively be introduced into the hydraulically settable mixture by adding an easily oxidized metal, such as aluminum, magnesium, zinc, or tin into a hydraulic mixture that is either naturally alkaline, such as a cementitious or calcium oxide containing mixture, or one that has been made alkaline, such as those containing gypsum or another lower alkaline hydraulic binder. This reaction results in the evolution of tiny hydrogen bubbles throughout the hydraulically settable mixture. Adding a base such as sodium hydroxide to, and/or heating, the hydraulically settable mixture increases the rate of hydrogen bubble generation.

Finally, air voids can be introduced into the hydraulically settable mixture during the molding process by adding a blowing agent to the mixture, which will expand when heat is added to the mixture. Blowing agents typically consist of a low boiling point liquid and finely divided calcium carbonate. The calcium carbonate and blowing agent are uniformly mixed into the hydraulically settable mixture. The liquid blowing agent penetrates into the pores of the individual calcium carbonate particles, which act as points from which the blowing agent can then be atomized upon thermal expansion of the blowing agent.

During the molding or extrusion process, the mixture is heated while at same time it is compressed. While the heat would normally cause the blowing agent to vaporize, the increase in pressure temporarily prevents the agent from vaporizing, thereby temporarily creating an equilibrium. When the pressure is released after the molding or extrusion of the material the blowing agent vaporizes, thereby expanding or "blowing" the hydraulically settable material. The hydraulically settable material eventually hardens with very finely dispersed voids throughout the structural matrix. Water can also act as a blowing agent as long as the mixture is heated above the boiling point of water and kept under pressure.

Air voids increase the insulative properties of the insulation barriers and also greatly decrease the bulk specific gravity, and hence the weight, of the final product. This reduces the overall mass of the resultant product, which reduces the amount of material that is required for the manufacture of the containers and reduces the mass of material that will eventually be discarded in the case of disposable containers.

H. Set Accelerators.

In some cases it may be desirable to accelerate the initial set of the hydraulically settable mixture by adding to the mixture an appropriate set accelerator. These include Na_2CO_3, KCO_3, KOH, NaOH, $CaCl_2$, CO_2, triethanolamine, aluminates, and the inorganic alkali salts of strong acids, such as HCl, HNO_3, and H_2SO_4. In fact, any compound which increases the solubility of gypsum and $Ca(OH)_2$ will tend to accelerate the initial set of hydraulically settable mixtures, particularly cementitious mixtures.

The amount of set accelerator which may be added to a particular hydraulically settable mixture will depend upon the degree of set acceleration that is desired. This in turn will depend on a variety of factors, including the mix design, the time interval between the steps of mixing the components and molding or extruding the hydraulically settable mixture, the temperature of the mixture, and the identity of the set accelerator. One of ordinary skill in the art will be able to adjust the amount of added set accelerator according to the parameters of a particular manufacturing process in order to optimize the setting time of the hydraulically settable mixture.

I. Coatings.

The sheets and containers that employ the hinges of the present invention may also have sealing materials or other protective coatings applied to their surfaces. Most coatings are applied with a solvent so that upon evaporation of the solvent the coating material remains on the surface of the sheet or container. One such coating is calcium carbonate, which also allows the printing of indicia on the surface of the sheets or containers.

Other coatings that can be used include melamine, polyvinyl chloride, polyvinyl alcohol, polyvinyl acetate, polyacrylate, hydroxypropylmethylcellulose, polyethylene - glycol, acrylics, polyurethane, polyethylene, polylactic acid, Biopol® (a polyhydroxy-butyrate-hydroxyvalerate copolymer manufactured by ICI in the United Kingdom), waxes (such as beeswax or petroleum based wax), elastomers, kaolin clay, polyacrylates, synthetic polymers including biodegradable polymers, ceramics, and mica.

Another type of coating that may be placed on the surface of the cementitious containers of the present invention is a reflective coating such as metal flake coatings for reflecting heat into or out of the container. Such reflective coatings are readily available, but their applicability to cementitious containers is novel.

In some cases, it may be preferable for the coating to be elastomeric, deformable, or waterproof. Some coatings may also be used to strengthen places where the

hydraulically settable sheets are more severely bent, such as where the sheet has been scored. In such cases, a pliable, possibly elastomeric, coating may be preferred. Besides these coatings, any appropriate coating material would work depending on the application involved.

For example, a coating comprised of sodium silicate, which is acid resistant, is a particularly useful coating. Resistance to acidity is important, for example, where the container is exposed to foods or drinks having a high acid content, such as soft drinks or juices. Where it is desirable to protect the container from basic substances, the containers can be coated with an appropriate polymer or wax, such as are used to coat paper containers. If the sheets are used to manufacture containers or other products intended to come into contact with foodstuffs the coating material will preferably comprise an FDA-approved coating.

The coatings may be applied to the sheets using any coating means known in the art of paper or cardboard making. Coatings may be applied by spraying the sheet, container, or other object with any of the above coating materials, or it may be advantageous to apply the coating by dipping the sheet, container, or other object into a vat containing an appropriate coating material. In the case where a coating material is sprayed onto the surface of a sheet, the coating material may be spread or smoothed by means of a doctor blade which is held a particular distance above the sheet, or which rides directly on the sheet surface.

In addition, coatings may be coextruded along with the sheet in order to integrate the coating process with the extrusion process. In other cases, the coating can be applied to the surface of the sheet by means of a gravure roller, often in conjunction with a doctor blade in order to smooth or adjust the thickness of the coating.

II. HINGES.

It is well known that paper products can bend easily and that traditional cement products do not bend much before fracture. The hydraulically settable materials that can be utilized in the present invention have greatly improved bendability prior to fracture because these materials can be made much thinner, while still holding form, than prior hydraulically settable materials. As discussed in more detail below, a score, perforation, or preformed bend is made in the hydraulically settable material to produce a hinge integrally formed into the material. The purpose of the score or perforation is to create a location on the hydraulically settable sheet where the sheet can be bent or folded. This

creates a "hinge" within the sheet with far greater bendability and resilience than possible with an unscored or unperforated hydraulically settable sheet.

A certain amount of tensile elongation is needed in order to give the hydraulically settable material bendability. The elongation of the material is greater than in typical hydraulic materials. Elongation is defined as the increase in length of a bar or section of material under test expressed as a percentage difference between the original length and the length at the moment of rupture. Expressed in a formula, percent elongation equals $100(L_f-L_o)/L_o$, wherein L_f is the final length at fracture and L_o is the initial length at rest. Typically, a material cannot exceed its percentage elongation without breaking.

In the present invention, the percent elongation of the hydraulic material matrix increases as the thickness of the material decreases. Conversely, as the thickness of the material increases, the elongation decreases. The fibers utilized in the material contribute to the bending of the structural matrix by increasing the elongation of the matrix before fracture. As more fibers are utilized, the percent elongation of the material is increased.

Another way to express the bending properties of the hydraulically settable material is through the bending radius of the material. The minimum bending radius (R) equals the elasticity modulus (Eo) times the thickness of the material (t) divided by two times the tensile strength (TS) of the material. This can be expressed in the following equation: $R = Eo(t)/2(TS)$. This means that if more fibers are put into the material, the tensile strength goes up and the elasticity goes down, so the material can bend in a very small radius. When the material has a higher elasticity, then a higher tensile strength is needed to bend the material in a desired manner or the thickness of the material must be decreased. The hinge of the present invention may be a living hinge or a nonliving hinge. A living hinge may be bent several times without breakage or fracture of the material. However, a nonliving hinge can be bent only once before breakage. A nonliving hinge of the present invention would have a low fiber content with a coating preferably over the surface of the hinge to hold it together until it is bent. The coating may be sprayed onto the hinge so that when the hinge is actually bent, the material of the hinge would be deformed underneath the coating and the coating would hold the material together after bending.

Coatings can be applied to the surface of the sheet to make it more flexible and can be applied to permanently soften the sheet or a hinge formed within the sheet. Elastomer, plastic, or paper coatings can aid in preserving the integrity of the hinge whether or not the underlying hardened structural matrix fractures upon bending at the

hinge. When the hinge is bent back, the material underneath the coating will bend or break. The only thing intact would be the coating on the surface.

The present hinge may also be designed so that it bends any number of different times before breaking, preferably 1 to 15 times. The hinge may also be perforated so that the material may be easily torn apart at the hinge location.

The materials utilized in the hinge may be varied in amounts and components in order to provide preselected properties for the hinge. For example, the fiber content utilized may be varied so that the hinge is not as flexible if a limited number of bends is desired in the product utilizing the hinge. Also, a variety of different fibers can be utilized and the fibers can be aligned, concentrated or consistently spread out. The fibers can also be co-extruded with the hydraulically settable material. Further, the thickness of the hinge may be altered or perforations may be put in the hinge matrix. All of the above may be altered in various combinations to provide specific characteristics in the hinge.

One way of making the hinge of the invention is by scoring the hydraulically settable material along a line after it has been formed into a sheet. Scoring is a process that displaces a given amount of material over a certain area by cutting or indenting the material with a steel plate or other device along a line, which aids in forming a bend or hinge at a predetermined location within the sheet. The score forming the hinge of the invention allows the hydraulic sheet material to be bent along the score up to about 180° from horizontal without fracture. The sheet preferably bends away from the score , which is different from paper-based materials that bend toward the score .

Furthermore, the hinge area of the hydraulically settable sheet below the score actually becomes stronger as a result of the densification of the hydraulically settable material at the score . This densification of the material below the score line is depicted in Figures 1-3, which show cross sections of hydraulically settable sheets having various scores that are discussed in further detail below.

The hinge of the invention can be advantageously formed during the sheet manufacturing process by scoring or perforating the sheet as it is being formed or soon thereafter. A score can also be cut into the surface of the sheet anytime after it is formed in order to create a line within the structural matrix upon which the sheet can later be bent.

Thus. the score can be made in the sheet while it is in the green state before the sheet is dry (e.g., while almost wet), in a semi-hardened state, or after it has become fully dried in order to form the hinge of the invention. For example. a flat sheet can be scored

and formed into the shape of a container and then hardened, or can be allowed to harden and then scored and formed into the shape of a container. The time and location of the placement of a score or perforation will depend upon the desired purpose thereof and the properties of the hydraulically settable material in question.

The hydraulically settable sheet will preferably be in a substantially form stable state during the scoring or perforation process. This is desirable to prevent the score or perforation from closing up through the migration of moist material thereinto . Since scoring generally (and perforation always) involves cutting through a portion of the structural matrix, the sheet can even be totally dry without the scoring or perforation process harming the sheet. In cases where a score is pressed rather than cut into the sheet surface, the sheet should be moist enough to prevent fracture due to the dislocation of the structural matrix.

The depth of the score cut will generally depend on the type of score, the thickness of the hydraulically settable sheet, and the degree of bending along the score line. The scoring mechanism should be adjusted to provide for a score of the desired depth. Of course, the die tap should not be so large as to actually cut through the sheet or render the sheet too thin to withstand the anticipated forces (unless an easily tearable score is desired). Preferably, the score cut should be just deep enough to adequately serve its purpose. A combination of score cuts on alternate sides of the sheet may be preferred in some cases to increase the range of bending motion.

In most cases where a thinner sheet (<1 mm) is being score cut or pressed, the score will have a depth relative to the thickness of the sheet that is within the range from between about 10% to about 50%, more preferably within the range from between about 20% to about 35%. In the case of thicker sheets, the score cut will usually be deeper due to the decrease in bendability of the thicker sheet. Thicker sheet materials can be scored to a depth up to about 90% of the total thickness of the material. In other words, the thickness of the hinge material after scoring can be as small as 10% of the original thickness of the sheet.

Scoring makes the remaining cross section very thin, with a preferred thickness of the material forming the hinge being in the range of about 0.01 to 1 mm, and a most preferred thickness in the range of about 0.05 to 0.5 mm. The material surrounding the hinge may be any thickness but the scored portion forming the hinge is very thin so that it may bend without breaking.

The hinge of the invention formed by scoring can have a variety of shapes in that the score line side view profile can be square, rectangular, rounded, parabolic, sinusoidal,

wedge, triangular, etc. The various shapes of the hinge design provide specific bending properties. Examples of the hinge design of the invention are depicted in Figures 1-3, showing that the hinge can be a single score , a double score , or a multiple score.

Figures 1A, 1B, and 1C show three hydraulically settable sheets 10, 12, and 14 in cross section side views having single scores 11, 13, and 15, respectively, of different shapes. Score 11 has a triangular profile, score 13 has a rectangular profile, and score 15 as a rounded profile. Figures 2A, 2B, and 2C depict three sheets 20, 22, and 24 in cross section side views having double scores 21, 23, and 25, respectively, of different shapes. Score 21 has a triangular profile, score 23 has a rectangular profile, and score 25 has a rounded profile.

Figures 3A-3F show six hydraulically settable sheets 30, 32, 34, 40, 42, and 44 in cross section side views having multiple scores 31, 33, 35, 41, 43, and 45, respectively, of different shapes. Scores 31 and 41 have a triangular profile, score 33 has a rectangular profile, scores 35 and 43 have a rounded profile, and score 45 has a sinusoidal profile.

Figure 3G depicts a hydraulically settable sheet 46 in a cross section side view having multiple scores 48 with rounded profiles on both sides of the sheet. This multiple scoring on both sides of sheet 46 enables sheet 46 to be bent along the score up to about 360° without fracture. Thus, multiple scoring on both sides of a hydraulically settable sheet allows the sheet to be bent in half in either direction.

When the sheet is scored on one side, such as shown in Figures 1 and 3, the sheet is preferably bent away from the score. For example, if the score is made in the top of the material, then the material is bent downwards. The bend is opposite the score such as shown in Figures 4 and 9. Double scoring of the sheet such as shown in Figure 2 provides a mechanism whereby the sheet can be bent in either direction without breaking. In double scoring, the scores are preferably made on both sides of the sheet as shown in Figure 2 to provide bending both ways if desired.

It should be understood that while the hydraulically settable sheets of the present invention will bend away from a score cut or perforation, the sheets will bend toward a score that is pressed into the surface of the sheet. Thus, the sides of the sheet defined by a score cut or perforation will close together on the side opposite the score cut or perforation as shown in Figures 4 and 9. Conversely, like paper or cardboard products, the sides of the hydraulically settable sheet defined by a score pressed into the sheet surface, as shown in Figure 7, will close together on the side of the score.

Multiple scoring of the sheet such as shown in Figure 3 provides increased bendability (compared to single scoring) without breakage or fracturing of the sheet.

This is shown in Figure 4A which depicts sheet 50 having single score 57, and in Figure 4B which depicts sheet 54 having multiple scores 55. Sheet 50 is shown bent at a maximum angle α which is less than the maximum angle β of sheet 54 having multiple scores. Multiple scores also allow sheets of a greater thickness to bend a greater amount, since each scored area of the sheet only has to bend a limited distance to provide the overall bending of the sheet. Multiple scores also provide for less stress on each scored area as the sheet is bent.

The scores are preferably integrally formed in the sheet with a die which stamps the score into the sheet formed of hydraulically settable material. The die used in forming the score has the same profile shape as the specific score made such as square, rectangular, rounded, parabolic, sinusoidal, wedge, triangular, etc.

It may sometimes be preferable to concentrate more fibers in the area where the score cut or perforation will be made to give greater strength and flexibility in the scored area which will be subject to bending. This can be accomplished by co-extruding a second layer of hydraulically settable material containing a higher fiber content at predetermined timed intervals to correspond with the location of the score cut or perforation. In addition, fibers can be placed on top of, or injected within, the sheet during the extrusion or calendering processes in order to achieve a higher fiber concentration at the desired location.

The hinge of the invention can be formed at a 0° angle or parallel with the direction of the fibers in the sheet, or can be formed at various angles up to 90° or perpendicular to the direction of the fibers. When the hinge is formed parallel to the fibers the hinge is weak. Such a hinge has minimum strength and maximum flexibility. When the hinge is formed perpendicular to the direction of the fibers, the hinge is strong. This hinge has maximum strength and minimum flexibility. Thus, the desired strength and flexibility of the material at the hinge location can be optimized when a hinge is formed at varying angles between 0° and 90° to the direction of the fibers. For example, scoring of the sheet to form a hinge is preferably done at a predetermined angle to the machine direction of the fibers within the sheet in order to optimize strength and flexibility of the hinge.

The act of forming or densifying of the hydraulically settable matrix correspondingly increases the density of the fibers in the area, preferably without damaging the fibers. In conventional tree paper, the act of scoring weakens the fibers therein to cause them to bend. In contrast, scoring of a hydraulically settable material causes the fibers beneath the score line to densify in the material and thus strengthens the

bend area of the material. For example, if the material has about 20% by volume of fibers, the area below a score line can increase up to about 40% by volume if the score depth is about 50% of the material thickness because of the densification of the material below the score line.

A score or perforation can be made on the hydraulic material sheet through the processes shown in Figures 5-8 in order to define a line upon which the sheet may fold or bend. As shown in Figure 5, a score cut 68 can be made on sheet 69 by using a knife blade cutter 70 mounted on a score press (not shown). As shown in Figure 6, a score cut 72 can be made on sheet 73 by using a continuous die cut roller 74. A score 76 may be pressed into sheet 77 by using a scoring die 78 as shown in Figure 7. The pressed score is made at a controlled rate, depth, and pressure when the sheet is in a wet or semi-dry condition. If the sheet is flexed at the score while wet a living hinge is formed. Perforations 80 can be made in sheet 81 using a perforation cutter 82 as depicted in Figure 8.

A score or perforation within the sheet creates a better fold line or hinge for a number of reasons. First, a score provides a place where the sheet might more naturally bend or fold. Second, a score makes the sheet at the score thinner than the rest of the sheet, which reduces the amount of lengthwise elongation of the surface while bending the sheet. The reduction of surface elongation reduces the tendency of the structural matrix to fracture upon being folded or bent. This is shown in Figure 9, which depicts sheet 84 bent along score cut 85. Third, the score cut or perforation allows for a controlled crack formation or failure within the matrix in the event that fracture of the structural matrix occurs.

The hinge of the invention can be used on a variety of different containers made from hydraulically settable materials which require a pivoting mechanism to open and close the container a number of times. An example of such a container is the "clamshell" container 90 depicted in Figures 10-12. Container 90 has upper shell member 92 which is pivotally attached to a lower shell member 93 by a hinge 94. Hinge 94 can be formed during the manufacture of container 90 by scoring the material forming container 90. Container 90 is used to store fast food items such as hamburgers or other sandwiches after they have been made and are delivered to the customer.

The hinge of the invention may include a pulp containing material such as a pulp sheet or paper which has been applied to the desired hinge area. The pulp sheet reinforces the hinge area by adding more fiber thereto and provides easier bending of the hinge area. This permits less fiber to be used in the rest of the matrix, if desired. The

pulp sheet also acts as a protective covering over the hinge area, eliminating dust from breakage of the material in the hinge area if this occurs.

Preferably, a paper strip is applied to the material forming the hinge area during the molding operation that forms the container. The paper strip can have a thickness as low as 0.01 mm. The paper strip is preferably fed into the molding machine perpendicular to the flow of hydraulic material. The molding force adheres the paper strip to the hydraulic matrix of the hinge area. In a preferred embodiment, the paper is applied to the inner side of the hinge area and a score is made on the outer side of the hinge area. This is shown in Figure 12, which depicts clamshell container 90 in an open position, with paper strip 95 disposed on the inner side of hinge 94, which pivotally connects shell members 92 and 93.

In an alternate embodiment, a paper laminate may be applied to the entire inner surface of the container, providing fiber reinforcement to the inside of the container. This provides flexibility and toughness to the container, as well as reinforcing the hinge area. The paper laminate itself can serve as the hinge, providing flexibility and a very high fiber content in the hinge area, and reinforcement of the remaining part of the container. This allows less fiber to be used in the hydraulic material used to form the container.

It is also possible to make the hinge of the invention by prebending or flexing a hydraulic material matrix in a semi-dry condition where the hinge is to be formed. In this way, memory is being introduced into the material and a living hinge is formed. When the material is bent in a semi-dry condition, there is not total adhesion of the cement onto the fibers, so that by bending it in the predried condition the actual hinge mechanism is being shaped into the material. Fibers in the material are pulled out of the matrix slightly to weaken the bond which gives better toughness and flexibility so as to allow pivotal movement at the bend point of the material. When the material hardens, the bend point or hinge is remembered by the material so that it bends in the same place every time.

The hinge of the present invention can also be made by localized "creping" of the hydraulically settable sheet material, which provides for improved bending or folding of the material. The hydraulically settable sheets are creped much like ordinary tree paper in order to provide a highly extensible sheet that is capable of absorbing energy at sudden rates of strain, and to provide improved bending of the sheet. Conventional creping can be performed either at the wet press section of a paper machine (wet crepe) or on a Yankee dryer (dry crepe). Although the exact parameters of either a wet or dry creping process will differ between the hydraulically settable sheets of the present invention and tree paper, one of ordinary skill in the art will recognize how to adjust the creping process

in order to obtain creped hydraulically settable sheets. The creping process would be expected to work better as the fiber content of the sheets is increased, since increased fiber content facilitates the sealing of the pores in the material and increased hydrogen bonding of the fibers.

If high temperatures are desired to be utilized in conjunction with the hinge of the present invention, then the preferred material is aluminate cement in combination with glass fibers. Temperatures of use with this material can range from about 250° to 900° C.

A preferred hinge of the present invention has a cementitious structural matrix formed from a reaction product of a cementitious mixture comprising about 1 to 70 percent by volume of a hydraulic cement; about 0.1 to 30 percent by volume of a rheology-modifying agent; a fibrous material comprising individual fibers having an aspect ratio of at least about 10:1; about 5 to 70 percent by volume of an aggregate material; and water added in an amount to result in a water to cement ratio of about 0.1:1 to about 10:1. The cementitious structural matrix of the hinge preferably has a thickness of about 0.01 to 1 millimeter, preferably about 0.05 to 0.5 millimeter.

III. <u>CONTAINERS AND OTHER PRODUCTS</u>.

The term "sheet" as used in this specification and the appended claims is intended to include any substantially flat, corrugated, curved, bent, or textured sheet made using the methods described herein. The only essential compositional limitation is that the structural matrix of at least part of the sheet includes a hydraulically settable mixture having a hydraulic binder and water. The sheet may include other materials such as paper, organic coatings, ink, or other nonhydraulically settable materials in addition to the hydraulically settable portion.

The term "container" as used in this specification and the appended claims is intended to include any receptacle or vessel utilized for packaging, storing, dispensing, portioning or shipping various types of products or objects (including, but not limited to, food and beverage products). Examples of such containers include boxes, cups, clamshells, jars, bottles, plates, trays, cartons, cases, crates, dishes, egg cartons, lids, straws, envelopes, or other types of holders.

In addition to integrally formed hydraulically settable containers, containment products used in conjunction with containers are also intended to be included within the term "containers." Such products include, for example, lids, liners, partitions, wrappers, cushioning materials, utensils, and any other product used in packaging, storing,

shipping, portioning, serving, or dispensing an object within a container, or wrapping an object held within a container.

In addition to sheets and containers, any object that can be formed using the hydraulically settable sheets described herein are also within the scope of the present invention. These include, for example, model airplanes, toys, venetian blinds, rain gutters, mailing tubes, shirt packaging forms, and temporary car window shades.

A cementitious container apparatus of the present invention comprises a first member, a second member adjacent to the first member, and means for flexibly joining the first and second members so that the first and second members can be pivotally moved about the joining means relative to one another. The joining means such as a hinge allows the first and second members to be pivotally moved between a first position wherein the first and second members are in straight alignment with one another and a plurality of other positions wherein the first and second members form an angle in relation to one another. The first and second members preferably have a mechanical resistance to bending and elongation within a first range, and the joining means has an area of reduced mechanical resistance to bending and elongation within a second range that is less than the first range. The first and second members also have a thickness within a first range and the joining means has an area of reduced thickness within a second range that is less than the first range of thickness.

The containers which use the hinge of the present invention may or may not be classified as being disposable. In some cases, where a stronger, more durable construction is required, the container might be capable of repeated use. On the other hand, the container might be manufactured in such a way that it is economical to use it once and then be discarded. Such disposable containers can be readily discarded or thrown away in conventional waste landfill areas as an environmentally neutral material.

The containers using the hinge of the present invention can be characterized as being lightweight, yet retaining sufficient strength for the desired purpose. When cementitious compositions are used in forming the containers, these compositions will preferably have a bulk specific gravity of less than 2.0 g/cm^3, and in some cases, less than 1.0 g/cm^3. Typically, the cementitious containers will have a compressive strength to bulk density ratio in the range from about 0.5 MPa-cm^3/g to about 1000 Mpa-cm^3/g. In the preferred embodiments, the compressive strength to bulk density ratio has a range from about 3 MPa-cm^3/g to about 50 MPa-cm^3/g, with the most preferred range being from about 5 MPa-cm^3/g to about 10 MPa-cm^3/g.

The structural matrix within the molded containers will preferably have a thickness less than about 20 mm, more preferably less than about 5 mm, and most preferably less than about 1 mm. In certain embodiments the thickness could even be less than 0.1 mm, especially where a more paper-like material is preferred.

The hinge of the present invention may also be utilized in making storage boxes with interlocking flaps. Single sheets can be scored to form the hinge as well as multiple sheets used for liners in a box. The hinge may also be utilized in making envelopes, gameboards, cereal boxes, cracker boxes, rice boxes, etc.

Products which utilize an accordion type hinge can be made with the cementitious hinge of the present invention. This falls within the definition of a hinge as used herein since an accordion hinge can be bent. An accordion hinge goes up and down in a wave-type motion. In making an accordion hinge, a cementitious material can be embossed so that corrugations or waves are put into the material where desired. An accordion hinge may also be made by scoring the material so that waves are thin and thick in an alternating fashion. Preferably, there are 1 to 6 waves in the accordion hinge.

One product using an accordion hinge is an accordion file, which can be made using the hinge of the present invention. An accordion file uses a type of hinge that can be expanded or contracted depending on how many papers are filed inside. Flexible straws can also be made with the present hinge in an accordion shape, and flexible ducts can be made using the hinge of the present invention.

The phrases "mass producible" or manufactured in a "commercial" or "economic" manner are intended to refer to a capability of the sheets and containers described herein to be rapidly produced at a rate that make their manufacture economically comparable to sheets and containers made from other materials, such as paper, cardboard, polystyrene, or metal. The sheets and containers used in the present invention are formed from innovative compositions which solve the prior art problems of incorporating hydraulically settable binders into the matrices of products, and can be rapidly manufactured or formed by machine rather than by manual formation.

Food or beverage product containers made from the hydraulic sheets used in the present invention are intended to be competitive in the marketplace with food or beverage containers currently made of various materials such as paper, plastic, polystyrene, or metals. Hence, the sheets and containers used in the present invention must be economical to manufacture (*i.e.*, the cost usually does not exceed a few cents per container). Such cost restraints thus require automated production of thousands of the articles in a very short period of time. Hence, requiring the products used in the present

invention to be economically mass-producibly manufactured is a significant limitation on the qualities of the products.

A significant advantage of the containers and hinges of the present invention is that they do not require or result in the emission of environmentally harmful or ozone depleting chemicals. In addition, when disposed of into the earth, such containers do not persist in the environment as do foreign materials which must biodegrade (often over a number of years) before they become environmentally innocuous. Instead, the waste containers are essentially composed of the same materials already found in the earth. Under the weight and pressure of typical landfills, such containers will crumble and break down into an environmentally neutral powder that is the same as, or at least compatible with, the dirt and rock already found within the landfill.

Furthermore, the containers are fully recyclable with a minimum amount of energy and effort. Unlike paper and plastic products, which require a substantial amount of processing in order to restore them to a suitable state as raw starting materials, hydraulically settable products can be ground up and recycled by merely reincorporating the grindings into new containers as an aggregate component within a hydraulic paste.

IV. PROCESSING METHODS AND TECHNIQUES.

The process for forming the containers and hinges of the invention with hydraulically settable materials can use a variety of methods which have been applied to plastic materials. The term "molding" as used herein includes the variety of molding, casting, and extrusion processes discussed herein or that are known in the art. These methods include roller casting, high pressure extrusion, ram pressing, hot isostatic pressing, injection molding, thermoforming, and other casting and forming methods.

The hinge of the present invention can be made in hydraulically settable sheets or containers having properties similar to those of paper, plastic, or thin-walled metals. The sheets can be immediately used to form a variety of objects such as food or beverage containers, or can be stacked or rolled and stored for future use. During the subsequent process of forming the sheet into the shape of the desired object, it will usually be advantageous to remoisten the hardened sheet in order to temporarily increase the flexibility and workability of the sheet to avoid splitting or cracking when the object is formed. This is particularly true in the case where the sheet will be rolled or has been scored and is expected to make a particularly sharp bend during the container forming stage.

The combination of high bulk and mass along with slow setting times has heretofore made it impossible to mass produce hydraulically settable sheets used to manufacture useful containers or other objects. While some relatively lightweight hydraulically settable products have been made (in very special settings in the construction industries), these products do not possess adequate tensile strength to be useful in making sheets having properties similar to those of paper, polystyrene, or other materials presently used to make containers or other products. Such prior art lightweight concretes are relatively weak, brittle, and cannot be formed into thin-walled objects having high tensile and compressive strengths, low overall mass, small size, and low cost.

The underlying theory behind the present invention is the rapid, continuous, and economical formation of lightweight, inexpensive sheets from a moldable hydraulically settable mixture, which sheets can be easily handled and rapidly manipulated in a commercial manufacturing setting much like paper, cardboard or plastics. The result is the ability to mass-produce in a very cost competitive manner lightweight, thin-walled, form stable sheets, as well as containers or other objects made therefrom, having a structural matrix that includes a hydraulically settable binder such as hydraulic cement or gypsum, rather than a paper, plastic, or polystyrene structural matrix. The combination of hydraulic material, water, fibers, and other materials results in a composition that can be formed into relatively thin sheets or walls having roughly the same thickness as conventional containers made from paper or styrofoam.

A method of making a hinge having a hydraulically settable matrix comprises the steps of mixing a hydraulically settable binder, fibers, and water in order to form a hydraulically settable mixture; forming the hydraulically settable mixture into a form stable sheet of a predetermined thickness; and scoring the sheet to form a hinge in the hydraulically settable matrix.

A preferred method of preparing a hydraulically settable material for use in making the container and hinges of the present invention includes the steps of mixing a powdered hydraulic material such as cement and water in order to form a hydraulic paste; combining a fibrous material with the paste under a high shear mixer to form a hydraulic mixture in which the fiber is well dispersed; adding a rheology-modifying agent so that the resultant mixture develops a more plastic-like rheology; and adding an aggregate or other material to the mixture using a low shear mixer in order to impart the desired lightweight properties to the mixture. Containers formed using the hydraulic mixture prepared by the above method typically have a bulk specific gravity of less than 1.5.

A high shear mixer is preferably used for the addition of fibrous material to insure that the fibers are well dispersed throughout the hydraulic material. To assist in the production of lightweight containers, it is also important to use standard low shear mixing for the addition of a lightweight aggregate such as perlite. This is to insure that the light weight aggregate is not crushed which in turn would increase the density of the aggregate and the resulting container.

A preferred method of making a hinge having a cementitious structural matrix comprises the steps of (1) mixing a hydraulic cement, water, a rheology-modifying agent, and a fibrous material to form a cementitious mixture; (2) molding the cementitious mixture into a predetermined shape, the mixture having adequate green strength to retain its shape without external support after being molded; and (3) scoring the molded cementitious mixture to form a cementitious hinge in the cementitious structural matrix.

Figure 13 shows a schematic diagram of a process for molding containers made of hydraulically settable materials that can utilize the hinge of the invention. The desired components such as hydraulic cement, water, fiber, plasticizers, aggregate, etc. are placed in a mixer 102 through an inlet 104 and are blended to form a hydraulic mixture. This hydraulic mixture is then directed to an extruder 106 where it is pumped through a die 108 into a roll-stack 110 that flattens the material into a sheet 112. The sheet 112 is then fed into a molding apparatus 114 to form the desired product such as a clamshell container. The formed sheet 116 is then directed through oven 118 to dry the material. The formed sheet 116 is then sent to a cutter machine 120 where the formed containers 122 are trimmed from formed sheet 116 and unloaded. The formed sheet 116 can be scored to form the hinge of the invention at anytime during the process of making the containers. For example, the hinge can be molded into the sheet between the parts of the molding apparatus when the container is formed therein.

In a preferred method of the above manufacturing process, the raw materials for making the cementitious mixture can be combined in a sigma-blade, kneading mixer system which will blend batches of material that are then deposited into the extrusion and calender system. Once the premixed material is deposited into the extruder, the material is auger fed into a chamber and extruded out through a rectangular slit about 42 inches wide and about 1/2 inch in height. The extruded material then passes through a series of heated rollers to produce a calendered sheet material, which reduces the web thickness to a height of about 2 mm. The rollers are heated to provide a mold release and will also partially cure the sheet material as water vapor is driven off. The sheet is preferably formed by pressing it between male and female molds using a vacuum forming apparatus.

A 64-cavity press can be used which operates at about 25 cycles/minute. Once the formed sheet is dried, it can be die cut in a punch press.

In order for the hydraulic material to exhibit higher flexural strength and/or cushioning, the fibers can be aligned or stacked instead of being randomly dispersed. It is preferable for the fibers to be laid out in a plane that is parallel to either of the two surfaces of the cement sheet or the container wall. In some containers, it is preferable for the fibers within the container body to be circumferentially or unidirectionally aligned. Similarly, the fibers within the bottom wall of the container can be horizontally aligned. Such alignment of fibers is achieved either by roller casting, ram-pressing, extrusion, or differential pressure roller extrusion.

By altering the quantities of hydraulic material, water, fibers, rheology-modifying agents, aggregates, etc. it is possible to control the rheology, or flow property, of the hydraulic paste. During the mixing of the paste, it is important to obtain flocculation or gelation of the mixture. In order for the hydraulic mixture to be effectively molded, it is important that the hydraulic material be form stable in the green state. The molded product should rapidly (preferably in three seconds or less) be able to support its own weight. Further, the material must harden sufficiently so that it can be quickly ejected from the mold. Otherwise, the cost of molding may make the process uneconomical. In addition, the surface of the molded article should not be too sticky, as that would make the demolding process difficult and cause problems in handling and stacking of the molded articles.

There are several modifications to conventional molding processes that can be employed in order to ease the manufacturing process of molding hydraulic materials. For example, it is frequently desirable to treat the mold with a releasing agent in order to prevent sticking. Suitable releasing agents include silicon oil, Teflon®, Deleron®, and UHW®. Preferably, the mold itself will be made of steel or of a material with a very "slick" finish, such as Teflon® or Deleron®. If the mold is made from steel, it will preferably be plated with either polished nickel or chromium. Regardless of the material used, it is important that the mold be kept hot (> 50° C) to create a thin layer of steam between the hydraulic mixture and the mold to aid in demolding the product. The same effect can be achieved from the use of frictional forces. By spinning the head of the molding apparatus against the interior and/or exterior surfaces of the hydraulic material, any chemical and mechanical adherence (*i.e.*, stickiness) to the mold can be overcome.

During the process of molding and/or curing the hydraulic mixture, it is often desirable to heat up the mixture in order to control the air void system and the porosity

of the molded container. This heating process also aids in making the hydraulic mixture form stable in the green state (immediately after molding) by allowing the surface to gain strength quickly. Heating also aids in rapidly removing significant amounts of water from the hydraulic mixture. The mixture can be heated to a temperature of about 50°C to 250°C during molding.

Although it is often preferable to coat the rollers with any of the nonstick materials discussed above, it is more important to heat the rollers to prevent sticking of the material to the rollers. Typically the roller temperatures will be within the range from about 50°C to about 150°C. Not only do the heated rollers prevent the hydraulic materials passed therethrough from sticking, but they also help the materials to more quickly reach form stability. The hydraulic mixture that is molded to form the containers and hinges of the invention is self-supporting during the green state and will maintain its formed state throughout the curing process. In addition, the hydraulic mixture rapidly reaches a sufficiently high strength so that the molded containers can be handled and manipulated using conventional means.

After the hydraulic mixture has been molded, it may be necessary to bend, fold, or otherwise shape the cured or uncured material into the desired shape of a container. For example, a flat sheet may be folded into the shape of a box. As the examples below demonstrate, it has been found that for some embodiments a box is most easily formed from a flat sheet containing fibers which is scored and folded after the sheet has been cured.

A method of manufacturing a bendable sheet having a hydraulically settable matrix comprises the steps of mixing a hydraulically settable binder, fibers, and water in order to form a hydraulically settable mixture; extruding the hydraulically settable mixture through a die; forming the extruded mixture into a form stable sheet of a predetermined thickness; and hardening the sheet to a significant degree in an accelerated manner in order to quickly increase the yield stress of the hydraulically settable matrix. The sheet can be made bendable by cutting a score, pressing a score, or cutting a perforation into a surface of the sheet that is substantially dried.

V. MANUFACTURING SHEETS FROM HYDRAULICALLY SETTABLE MIXTURES.

The sheet used in the present invention is formed by a sheet and calendering process in which an appropriate hydraulically settable mixture having the desired characteristics is first extruded through a die using, for example, an auger- or piston-type

extruder, into thin sheets (or similar shapes) of a predetermined thickness. The extruded sheets are then "calendered" by passing them between at least one pair of rollers to form sheets with reduced but more uniform thickness and a generally smoother surface. The term "calender" refers to the process of passing the hydraulically settable material between one or more sets of rollers in order to reduce the thickness and/or finish the surface and/or dry the surface of the resulting sheet. Because the manufacturing processes described by the present invention do not necessarily require that each of these subprocesses be performed by rollers, the term "calender" can refer to any of these subprocesses alone or in combination with either or both of the other subprocesses.

If a series of rollers are used, the roller pairs have successively narrower gaps between them in order to create a progressively thinner sheet. The extrusion process in combination with the elongation of the sheet during the calendering process tends to orient the fibers in the "Y" direction. Conically shaped rollers can be used in order to widen the sheet and also orient some of the fibers in the "X" direction. In this way, it is possible to obtain a sheet with bidirectionally oriented fibers.

The rollers are preferably treated in order to prevent adhesion between the sheet and the rollers. This may be accomplished by coating the rollers with a nonstick substance, polishing the rollers, heating the rollers to form a steam barrier, cooling the rollers to form a condensation barrier or a combination of these. Heating the rollers also has the often desirable effect of driving off significant amounts of excess water within the sheets.

The calendered sheets are substantially or completely dried by passing the sheets through a series of individual rollers, through one or more pairs of rollers, passing the sheet through a drying tunnel, or a combination thereof. The removal of water from the sheets imparts higher green strength and form stability, while the heat used to remove the water accelerates the rate of hydration or curing of the hydraulic binder. By significantly raising the temperature, it is possible to obtain substantially full strength development within a day or two after first mixing the hydraulically settable material.

After a significant portion of the water has been removed from the formed sheet, the sheet can optionally be compressed to remove unwanted voids within the structural matrix of a semi-hardened sheet, which densifies and strengthens the sheet. This is performed by passing a semi-dried sheet through at least one pair of compaction rollers. The moisture content of the sheets just prior to compaction should be carefully controlled in order to ensure that the sheets are compacted rather than elongated as in the reduction process. If the sheets are compacted while in a semi-dry condition, they may then be

further dried using the same methods and apparatus described above for the first drying step.

In addition, the sheet can be compacted to the final thickness and "finished" by passing the sheet through at least one pair of finishing rollers consisting of a hard and soft roller. The "hard" roller is a highly polished roller which imparts a high degree of smoothness on the side of the sheet adjacent to the hard roller. The "soft" roller is an un-polished metal roller with enough friction to pull the sheet between the finishing rollers with adequate tension (the smooth roller being essentially frictionless and unable to pull the sheet through the rollers) to cause the finishing rollers to slip over and slide over the surface of the sheet to create a smoother surface. Alternatively, a clay coating can be sprayed onto the sheet so that the finishing rollers smooth the surface using the coating.

Finally, the semi-hardened or hardened sheets can then be used much like paper or cardboard to manufacture a variety of containers, printed materials, or other objects, or they can be rolled onto a spool or cut and stacked onto a pallet and stored until needed. The sheets can be scored, score cut, or perforated in order to create a fold line, then folded and/or rolled into the desired shape of the container or other object. When folding or rolling the sheet, it will often be advantageous to remoisten the sheet in order to introduce temporary increased flexibility.

The rolled and/or folded sheet in the desired shape of the container or other object can be held together using any connection means known in the art. In some cases, the ends can be folded together or inserted into specially designed slots. Alternatively, the adjacent hydraulically settable surfaces can be subjected to high pressure to form a single thickness of material, thereby sealing or bonding the surfaces together. In other cases, it may be necessary to glue the corresponding ends together using adhesion means known in the art. These include glue, adhesive strips, thermoplastic materials, or a combination thereof. The hydraulically settable sheets, containers, or other objects can be coated with a desired coating or printed on using means known in the art of paper, plastic, or polystyrene use. This can be done at any appropriate stage of the manufacturing process.

A comprehensive production sequence for a hydraulically settable sheet useful in the present invention is set forth in Figure 14, including apparatus for carrying out the following manufacturing steps: (1) mixing the hydraulically settable mixture; (2) extruding the mixture into a flat sheet, or other object through an appropriate extruder die; (3) calendering the extruded sheet by passing it through a series of paired rollers in order to reduce the thickness and improve the surface qualities of the sheet; (4) at least partially drying the sheet by rolling it onto one or more rollers; (5) optionally compacting

the sheet while in a slightly moist condition in order to eliminate unwanted spaces within the structural matrix of the sheet and to increase the density and resulting strength of the sheet; (6) optionally drying the sheet after it has been compacted; (7) optionally finishing the sheet by passing it between one or more pairs of rollers, including one hard and one soft roller; and (8) optionally rolling the substantially hardened and dried sheet onto a spool to form a roll that can be stored and used when needed.

In the case where the hydraulic mixture is extruded into any object other than a sheet, it will often be necessary to "open up" the object into a sheet (such as continuously cutting a pipe to form a sheet). If another shape is extruded, other procedures (such as the use of additional rolling processes) may need to be employed. However, the same principles described herein would apply to other extruded shapes. Each of these manufacturing steps is set forth more fully hereinbelow.

A. Preparing The Hydraulically Settable Mixture

The first step in the manufacture of sheets involves the formation of a suitable hydraulically settable mixture having the desired workability, green strength, and final properties after hardening. Using a microstructural engineering approach, one skilled in the art can select the components, as well as their relative concentrations, in order to obtain a hydraulically settable mixture having the desired properties.

Some of the properties considered to be generally desirable with regard to the hydraulically settable mixture are adequate workability, plastic-like qualities, and green strength for a given extrusion, rolling, and/or molding process. As set forth above, the level of water, rheology-modifying agent, and (optionally) dispersant will determine the level of workability and extrudability of the mixture, as will the other components within the mixture, such as aggregates, fibers, set accelerators, etc. Based on the teachings of the copending applications incorporated herein by reference, one skilled in the art will be able to adjust the identities and amounts of the various components in order to optimize the workability, plastic-like behavior, and green strength necessary to carry out any particular sheet forming process.

With regard to the final cured or hardened product, some of the properties considered generally desirable to design into the structural matrix of the sheet include high tensile strength (in general or along particular vectors), flexural strength, flexibility, and ability to elongate, deflect or bend. In some cases it may be desirable to obtain sheets which substantially incorporate the properties of existing paper or cardboard products. In other cases it may be desirable to obtain a structural matrix having properties not

obtainable using ordinary wood pulp or other traditional paper-making starting materials. These may include increased toughness, higher modulus, water resistance, or lower bulk density.

The flexibility, tensile strength, flexural strength, or modulus can be tailored to the particular performance criteria of the sheet, container, or other object in question by adjusting the components and relative concentrations of the components within the hydraulically settable mixture. In some cases, higher tensile strength may be an important feature. In others, it may be less significant. Some sheets should preferably be more flexible, while others will be stiff. The important thing is to achieve a material which has properties adequate for a particular use, while remaining cognizant of cost and other practical production line parameters. While having "too much" or "too little" of a particular property may be inconsequential from the standpoint of performance, from a cost standpoint it may be wasteful or inefficient to provide for the particular property.

The hydraulically settable sheets formed using the compositions described above will preferably have a tensile strength within the range from about 0.05 MPa to about 70 MPa, and more preferably within the range from about 5 MPa to about 25 MPa. In addition, the sheets will preferably have a bulk density within the range from about 0.1 g/cm^3 to about 2 g/cm^3. Whether a sheet will have a density at the lower, intermediate, or higher end of this range will generally depend on the desired performance criteria for a given usage. Finally, the hydraulically settable sheets of the present invention will preferably have a tensile strength to bulk density ratio within the range from between about 2 MPa-cm^3/g to about 70 MPa-cm^3/g, and more preferably within the range from between about 3 MPa-cm^3/g to about 25 MPa-cm^3/g.

The term "elongate" as used herein with regard to the hydraulically settable sheets means that the structural matrix of the sheets is capable of being stretched without rupturing and still have a finished surface. In other words, the structural matrix of the sheets is capable of moving or changing shape without rupture by application of a force such as pulling or stretching. The ability of the structural matrix to elongate before rupture is measured by an Instron tensile test and a stress-strain test.

By optimizing the mix design, it is possible to manufacture a sheet that has a structural matrix capable of elongating up to about 20% in the wet state before tearing or rupturing occurs, and from about 0.5% to 8% in the dry state. This is usually accomplished by optimizing the amount of fiber and/or rheology-modifying agent within the hydraulically settable mixture. Producing a sheet that has a structural matrix capable of elongating within the specified range can be accomplished by including fibers within

the hydraulic mixture in an amount up to about 50% by volume of the hydraulically settable mixture. When greater amounts of fibers or rheology-modifying agents are added, more elongation can generally be achieved without rupture to the sheet. In addition, the elongation of a dry sheet can be increased by adding steam or moisture to the sheet in the order of up to 10% by weight of the dry weight of the sheet. However, this remoistening reduces the ultimate strength of the sheet.

Rheology-modifying agents added to mixtures having fibrous materials can be added in an amount of up to about 50% by weight of the hydraulically settable mixture, and more preferably within a range from about 0.1% to about 10% by weight of the green hydraulically settable mixture. In an alternative embodiment where fibers are not included, the rheology-modifying agent will be included in an amount within the range from about 0.5% to about 5% by weight of the hydraulically settable mixture.

It should be understood that higher tensile strength, as well as greater elongation, will generally be obtained by increasing the amount of fibers within the structural matrix. This can be accomplished by adding more fibers to the hydraulically settable mixture or, alternatively, by attaching a layer of fibers (such as a sheet of paper) on the surface or within the interior of a hydraulically settable sheet.

The term "deflect" as used herein with regard to the hydraulically settable sheets means that the sheets have a structural matrix capable of bending and rolling without rupture and still have a finished surface. The ability of a sheet to deflect is determined by measuring the elasticity modulus and the fracture energy of the sheet using means known in the art. As with any material, the bending ability of a sheet manufactured according to the present invention improves with decreasing thickness of the sheet.

One way to measure deflection without regard to sheet thickness is to define deflection as the relative elongation of one side of the sheet compared to the other side of the sheet. As a sheet is rolled or bent around an axis, the length of the outer side of the sheet will elongate, while the inner side of the sheet generally will not. Consequently, a thinner sheet can be bent a far greater degree even though the relative elongation of the outer side compared to the elongation of the inner side is about the same as in a thicker sheet that cannot bend nearly as far. As a general rule, the degree of elongation of an outer side before fracture is independent of the thickness of the sheet although properties of bendability would be expected to increase as the thickness of the sheet decreases and approaches the thickness of the individual fibers within the structural matrix of the sheet.

As a general rule, a sheet within the scope of the present invention can be made that will have a structural matrix so that one side can elongate with respect to the other

side of the sheet. This ability of the sheet to deflect is related to the sheet's ability to elongate; consequently, the optimal mix designs for achieving the desired deflection range are similar to the mix designs for achieving the desired level of elongation. Nevertheless, during the process of forming the sheet into an appropriate food or beverage container or other object the bendability of the sheet can be temporarily increased by remoistening the sheet. The water is believed to be absorbed by the fibers, rheology-modifying agent, and the interstices between the cement and aggregate particles. Upon drying the formed sheet, the level of bendability will generally decrease while the rigidity and hardness of the sheet will generally increase.

In order to obtain a sheet having the desired properties of strength, bendability, insulation, toughness, weight, or other performance criteria, the thickness of the sheet can be altered by adjusting the space between the rollers, as set forth more fully below. Depending on the thickness and desired performance criteria, the components and their relative concentrations can be adjusted in order to accommodate a particular sheet thickness. The sheets of the present invention may be designed to have a thickness of a very wide range; however, most products using a thin-walled material will generally have a thickness in the range from about 0.01 mm to about 3 mm.

The preferred thickness of the sheets of the present invention will vary depending on the intended use of the hydraulically settable sheet, container, or object to be made. As a matter of example only, where high deflectability is desired, a thinner sheet will generally be preferred. Conversely, where strength, durability, and/or insulation and not deflectability are the overriding concerns, a thicker sheet will generally be preferred. Nevertheless, where it is desired to bend the sheets along a score, or at least roll them into containers, the hydraulically settable sheets will preferably have a thickness within the range from about 0.01 mm to about 2 mm, and more preferably within the range from about 0.03 mm to about 1 mm.

Another aspect of the present invention is the ability of the extruded and calendered material to have high green strength. This can be achieved by adding a rheology-modifying agent such as a polysaccharide- or protein-based material in order to increase the yield stress of the hydraulically settable mixture. A preferred polysaccharide additive is Tylose®, which is a commercial embodiment of methylhydroxyethylcellulose. Tylose® also creates a more plastic-like, workable material, which is believed to involve the bridging of the individual hydraulic binder particles by the Tylose®, which is adsorbed onto the surfaces of the particles.

Although it is preferable that the molded product have a relatively low water to cement ratio, in the initial mixing stage of the hydraulically settable mixture it is possible, and often desirable, to have a relatively high water to cement ratio, often as high as 4:1. This is because in the preferred method of molding the sheet, which is discussed more fully below, the hydraulically settable mixture is usually passed through a series of heated rollers which drive off a significant amount of water and aid in molding a sheet with high green strength. Nevertheless, one skilled in the art may adjust the water content so that the hydraulic mixture has an appropriate rheology so that it will be easily and effectively extruded through a particular die.

In order to prepare a desired hydraulically settable mixture, the fiber, water, rheology-modifying agent, and other additives are preferably blended together in a high shear mixer in order to form a well-dispersed, homogeneous mixture. High shear mixing is used for the addition of fibrous material to insure that the fibrous materials are well dispersed throughout the mixture. This results in a more uniformly blended mixture, which improves the consistency of the uncured mixture and increases the strength of the final cured product. It may be preferable to also add the hydraulic binder, as well as certain lower concentration aggregates such as mica, during the high shear mixing step in order to get a homogenous mixture in the shortest possible time.

The addition of fibrous materials by normal cement mixing techniques usually results in the conglomeration of the fibers, leading to deformities in the resulting sheets or articles. Standard mixers, such as drum mixers, combine the components of the desired mixture by applying low energy stirring or rotating to the components. In contrast, high shear mixers are capable of rapidly blending the mixture so as to apply high shearing forces on the particles within the hydraulically settable materials and the added fibrous materials. As a result, the fibrous materials and particles are uniformly dispersed throughout the mixture, thereby creating a more homogenous structural matrix within the hardened sheets. Fine particulate aggregates of relative high strength, such as sand, silica, or alumina, can also be blended using a high speed mixer, although not if included in such high concentrations as to cause the hydraulic mixture to have a relatively low water content and high viscosity.

Thereafter, aggregates included in higher concentrations (and sometimes the hydraulic binder) are blended into the mixture using a low shear mixer. This is particularly true where lightweight aggregates are added which cannot withstand high shear conditions without breaking, such as perlite or hollow glass spheres. It is preferable that the size of the aggregates not exceed about 30% of the final thickness of the sheet,

since oversized aggregates could damage the rollers and create flaws within the sheet surface.

Whether or not the hydraulic binder is added during the steps of high or low shear mixing depends on the nature of the hydraulic binder as well as how the mixture is handled. For example, it is believed that high shear mixing of hydraulic cement after the formation of a particulate hydrosol gel can disrupt the gel and result in a final hardened product with dramatically lower compressive and tensile strengths.

In alternative embodiments, other additives such as air-entraining agents and reactive metals can be incorporated into the mixture in order to obtain a final material with lower density and higher insulating ability.

In a typical mixing process in the laboratory, the appropriate components are blended using a high shear, high speed mixer for about 1 minute. Thereafter, the remaining components are blended into the mixture using a low shear, low speed mixer for about 5 minutes. The total mixing time per batch of material is therefore about 6 minutes, although this may be varied depending upon the nature of the hydraulically settable mixture. Industrially, this mixing time can be shortened by the use of appropriate mixers; specifically, the currently preferred method of mixing being a continuous mixing system.

In one embodiment, a cement mixer capable of both high and low shear mixing is used to meter and mix the materials in a batch mode. This mixer can handle up to 350 l. of material per batch and, assuming a 6 minute mix cycle, is capable of producing 2,000 Kg of a hydraulically settable mixture per hour, assuming 0.5 g/cm^3 per cubic foot.

In an alternative embodiment, high speed, high shear mixers described in United States Patent No. 4,225,247 entitled "Mixing and Agitating Device" and United States Patent No. 4,552,463 entitled "Methods and Apparatus for Producing a Colloidal Mixture," can be used for mixing the hydraulically settable mixture. Thereafter, the mixture can be transferred to a low speed, low shear mixer in order to complete the mixing process. The mixing step may also be combined with the extrusion step (discussed below) using modern extrusion equipment that includes a high shear mixing chamber.

The currently preferred embodiment for the industrial setting is equipment in which the materials incorporated into the hydraulically settable mixture are automatically and continuously metered, mixed, deaired, and extruded by a twin auger extruder apparatus. A twin auger extruder apparatus has sections with specific purposes such as low shear mixing, high shear mixing, vacuuming, and pumping. A twin auger extruder

apparatus has different flight pitches and orientations which permits the sections to accomplish their specific purposes.

It is also possible to premix some of the components in a vessel, as needed, and pump the premixed components into the twin auger extruder apparatus. The preferable twin auger extruder apparatus utilizes uniform rotational augers wherein the augers rotate in the same direction. Counter-rotational twin auger extruders, wherein the augers rotate in the opposite directions, accomplish the same purposes. A pugmil may also be utilized for the same purposes. Equipment meeting these specifications are available from Buhler-Miag, Inc., located in Minneapolis, Minnesota.

The internal components of the mixer can be made of stainless steel because the abrasion to the mixer is not too great in light of the high water content. However, the mixer components can be carbide coated for extended life, thereby resisting any abrasion and the strongly basic conditions expected from a mixture containing aggregates and a hydraulic cement.

The various component materials that are combined within the hydraulically settable mixtures of the present invention are readily available and may be purchased inexpensively in bulk quantities. They may be shipped and stored in bags, bins, or train cars, and later moved or unloaded using conventional means known in the art. In addition, the materials can be stored in large storage silos and then withdrawn and transported by means of conveyors to the mixing site.

As previously discussed, the hydraulically settable mixture is microstructurally engineered to have certain desired properties. Consequently, it is important to accurately meter the amount of material that is added during any batch or continuous admixing of the components.

B. Molding Sheets from the Hydraulically Settable Mixture.

Once the hydraulically settable mixture has been properly blended, the mixture is then transported to the sheet forming apparatus, which will typically comprise an extruder and a set or series of rollers. In some cases an apparatus which both mixes and extrudes the hydraulically settable mixture may be used in order to streamline the operation and minimize the coordination of the various components within the system.

Reference is now made to Figure 14, which illustrates a currently preferred system for manufacturing sheets from a hydraulically settable mixture. The system includes a mixing apparatus 150, an auger extruder 160, forming rollers 171, drying rollers 172, optional compaction rollers 174, optional second drying rollers 178, optional

calendering rollers 180, and optional spooler 182. In the first currently preferred sheet forming step, the hydraulically settable mixture can be formed into sheets of precise thickness by first extruding the material through an appropriate extruder die and then passing the extruded material through one or more pairs of reduction rollers as shown in Figure 14.

Figure 15 is an enlarged view of auger extruder 160 shown in the system of Figure 14. Auger extruder 160 includes a feeder 161 that feeds the hydraulically settable mixture into a first interior chamber 162 within the extruder 160. Within the first interior chamber 162 is a first auger screw 163 that exerts forward pressure on and advances the hydraulically settable mixture through the first interior chamber 162 toward an evacuation chamber 164. Typically, a negative pressure or vacuum will be applied to evacuation chamber 164 in order to remove unwanted air voids within the hydraulically settable mixture.

Thereafter, the hydraulically settable mixture will be fed into a second interior chamber 165. A second auger screw 166 will advance the mixture toward a die head 167 having a transverse slit 168 with a die width 169 and a die thickness 170. The cross-sectional shape of transverse slit 168 is configured to create a sheet of a desired width and thickness that will generally correspond to die width 169 and die thickness 170.

An important advantage of using an auger extruder is that it allows for a continuous extrusion process. In addition, an auger extruder has the ability to remove unwanted macroscopic air voids within the hydraulically settable mixture. Failure to remove unwanted air voids can result in the sheet having a defective or nonhomogeneous structural matrix. During subsequent drying steps, particularly where relatively high heat is used, unwanted air pockets can greatly expand and cause air bubble defects. However, such defects will generally not occur in the case where finely divided air voids are incorporated within the hydraulically settable mixture.

Removal of the air voids may be accomplished using conventional venting means known in the extrusion art as shown in Figure 15, wherein the mixture is first passed into an evacuation chamber 164 by first auger screw 163, and then extruded through the extruder die head 167 by means of second auger screw 166. Alternatively, the unwanted air voids may be removed from the hydraulically settable mixture by a process known as "venting" wherein the excess air collects under pressure within the interior chamber and escapes from the extruder by passing through the space defined by the walls of the interior chamber and the outer edges of the auger screw.

Although the preferred width and thickness of the die will depend upon the width and thickness of the particular sheet to be manufactured, the thickness of the extruded sheet will usually be at least twice, and sometime many times, the thickness of the final calendered sheet. The amount of reduction (and, correspondingly, the thickness multiplier) will depend upon the properties of the sheet in question. Because the reduction process helps control fiber orientation, the amount of reduction will often correspond to the degree of desired orientation. In addition, the greater the thickness reduction, the greater the elongation of the sheet. In a typical manufacturing process an extruded sheet with a thickness of about 6 mm may be calendered to a sheet with a thickness between about 0.2 mm and about 0.5 mm.

Although the die slit is generally rectangularly shaped, it may contain areas of increased thickness along its width in order to form an extruded sheet having varying thickness along its width. In this case, if rollers are used in conjunction with the extrusion process they will preferably have recesses or gap variations which correspond to the areas of increased thickness within the extruded sheet. In this way a sheet having reinforced areas of increased strength and stiffness can be produced.

In an alternative embodiment, it is envisioned that the width of the die slit can be selectively varied as a function of time as the mixture is extruded through the slit. This permits the extrusion of a sheet having varying thickness along the length of the sheet. In this scenario, it will generally be necessary to provide rollers that also have varying gap distances as a function of time. However, because of the greater difficulty of perfectly synchronizing the rollers to accommodate the rate of extrusion of sheets of varying thickness, this option is less preferable than creating a sheet with varying thickness along the width as described above.

In addition to narrow die slits to form flat sheets, other dies may be used to form other objects or shapes. The only criterion being that the extruded shape be capable of being thereafter formed into a sheet. For example, in some cases it may not be desirable to extrude an extremely wide sheet. Instead, a pipe may be extruded and continuously cut and unfolded using a knife located just outside the die head.

The amount of pressure that is applied in order to extrude the hydraulically settable mixture will generally depend on the pressure needed to force the mixture through the die head, as well as the desired rate of extrusion. It should be understood that the rate of extrusion must be carefully controlled in order for the rate of sheet formation to correspond to the speed at which the sheet is subsequently passed through the calendering rollers during the calendering step. If the rate of extrusion is too high, excess

hydraulically settable material will tend to build up behind the calendering rollers, which will eventually cause a clogging of the system. Conversely, if the rate of extrusion is too low, the calendering rollers will tend to stretch the extruded sheet, which can result in a fractured or uneven structural matrix, or worse, breakage or tearing of the sheet. The latter can also result in a complete breakdown of the continuous sheet forming process.

It will be understood that an important factor which determines the optimum speed or rate of extrusion is the final thickness of the sheet. A thicker sheet contains more material and will require a higher rate of extrusion to provide the necessary material. Conversely, a thinner sheet contains less material and will require a lower rate of extrusion in order to provide the necessary material.

The ability of the hydraulically settable mixture to be extruded through the die head, as well as the rate at which it is extruded, is generally a function of the rheology of the mixture, as well as the operating parameters and properties of the machinery. Factors such as the amount of water, rheology-modifying agent, dispersant, or the level of initial hydration of the hydraulic binder all affect the rheological properties of the mixture. Therefore, in order to control the rate of extrusion it will be preferable to carefully control the mix design and the rate of setting of the hydraulically settable mixture.

Because it will sometimes not be possible to control all of the variables that can affect the rate of extrusion, it may be preferable to have an integrated system of transducers which can measure the rate of extrusion or can detect any buildup of excess material behind the calendering rollers. This information can then be fed into a computer processor that can then send signals to the extruder in order to adjust the pressure and rate of extrusion in order to fine tune the overall system. A properly integrated system will also be capable of monitoring and adjusting the roller speed as well.

As set forth above, adequate pressure is necessary in order to temporarily increase the workability of the hydraulically settable mixture in the case where the mixture has a deficiency of water and has a degree of particle packing optimization. In a mixture that is water deficient, the spaces (or interstices) between the particles contain insufficient water to lubricate the particles in order to create adequate workability under ordinary conditions.

As the mixture is compressed within the extruder, the compressive forces force the particles together, thereby reducing the interstitial space between the particles and increasing the apparent amount of water that is available to lubricate the particles. In this way, workability is increased until the mixture has been extruded through the die head,

at which point the reduced pressure causes the mixture to exhibit an almost immediate increase in stiffness and green strength, which is generally desirable.

It should be understood that the pressure exerted on the hydraulically settable mixture during the extrusion process should not be so great as to crush or fracture the lightweight, lower strength aggregates (such as perlite, hollow glass spheres, pumice, or exfoliated rock). Crushing or otherwise destroying the structural integrity of these or similar lightweight aggregates containing a large amount of voids will decrease their insulating effect by eliminating the voids. Nevertheless, because perlite, exfoliated rock, or other such materials are relatively inexpensive, some level of crushing or fracturing of the aggregate particles is acceptable. At some point, however, excess pressure will eliminate the lightweight and/or insulative effect of the lightweight aggregate, at which point it would be more economical to simply include a less expensive aggregate such as sand.

In light of each of the factors listed above, the amount of pressure that will be applied by the extruder in order to extrude the hydraulically settable mixture will preferably be within the range from between about 50 kPa to about 70 MPa, more preferably within the range from between about 150 kPa to about 30 MPa, and most preferably within the range from between about 350 kPa to about 3.5 MPa.

In some cases, particularly where a lower density, higher insulating sheet is desired, it may be advantageous to employ a blowing agent that is added to the mixture prior to the extrusion process.

It will be understood that the extrusion of a hydraulically settable binder through the die head will tend to unidirectionally orient the individual fibers within the hydraulically settable mixture along the "Y" axis, or in the lengthwise direction of the extruded sheet. As will be seen herein below, the calendering process will further orient the fibers in the "Y" direction as the sheet is further elongated during the reduction process. In addition, by employing rollers having varying gap distances in the "Z" direction (such as conical rollers) some of the fibers can also be oriented in the "X" direction, *i.e.*, along the width of the sheet. Thus, it is possible to create a sheet by extrusion, coupled with calendering, which will have bidirectionally oriented fibers.

C. The Calendering Process.

In most embodiments, it will be preferable to "calender" the extruded sheet by passing it between at least one pair of rollers, the purpose of which is to improve the uniformity and surface quality of the sheet and also usually reduce the thickness of the

sheet. In some embodiments, the calendering step will only reduce the thickness of the sheet by a small amount, if at all. In other cases, the calendering process will substantially reduce the thickness of the sheet. In cases where it is desirable to greatly reduce the thickness of the hydraulically settable sheet, it will often be necessary to reduce the thickness of the sheet in steps, wherein the sheet is passed through several pairs of rollers, with each pair having progressively narrower gap distances therebetween.

Reference should be made to Figure 14 which shows one embodiment in which a series of pairs of rollers are employed during the calendering step. The rollers within each of the pairs have similar diameters, although in some cases it may be preferable to use smaller diameter rollers in combination with larger diameter rollers.

As the thickness of the sheet is reduced upon passing through a pair of rollers, it will also elongate in the forward moving (or "Y") direction. One consequence of sheet elongation is that the fibers will further be oriented or lined up in the "Y" direction. In this way, the reduction process in combination with the initial extrusion process will create a sheet having substantially unidirectionally oriented fibers in the "Y", or lengthwise, direction.

Another consequence of sheet elongation is that the sheet will "speed up" as it passes between a pair of reduction rollers. The consequence of this is that the roller speed will be "faster" relative to the speed of the sheet as it enters into the rollers. By way of example, if the sheet thickness is reduced by 50% and assuming there is no widening of the sheet during the reduction process the sheet will elongate to twice its original length. This corresponds to a doubling of the sheet's velocity before it enters the rollers compared to when it exits the rollers.

The sheet "speeds up" while passing between a pair of rollers by being squeezed or pressed into a thinner sheet by the rotating rollers. This process of squeezing or pressing the sheet, as well as the speed differential between the entering sheet and the rollers, creates a certain amount of shearing forces on the sheet. The application of an excessively large shearing force can disrupt the integrity of the structural matrix of the sheet and create flaws within the sheet, thereby weakening the sheet. Because of this, the thickness of the sheet should be reduced in steps small enough to prevent undue damage to the sheet. In light of typical production parameters (such as, e.g., minimizing the number of reduction steps, orienting the fibers, and controlling the rheology of the hydraulically settable mixture) the reduction in thickness of a sheet will preferably not exceed about 75% during any single reduction step (*i.e.*, while passing between any one

set of rollers), more preferably no greater than about 50%, and most preferably no greater than about 30%.

The diameter of each of the rollers should be optimized depending on the properties of the hydraulically settable mixture and the amount of thickness reduction of the hydraulically settable sheets. When optimizing the diameter of the rollers two competing interests should be considered. The first relates to the fact that smaller diameter rollers tend to impart a greater amount of shearing force into the sheet as the sheet passes between the rollers. This is because the downward angle of compression onto the sheet is on average greater than when using a larger diameter roller. Thus, larger diameter rollers would appear to be advantageous compared to smaller diameter rollers because less shearing forces would be expected to introduce fewer flaws into the hydraulically settable structural matrix.

The use of larger diameter rollers has the drawback of allowing the hydraulically settable material to come into contact with the roller for a greater period of time, thereby resulting in an increase in drying of the sheet during the calendering process in the case where the rollers are heated to prevent adhesion. Since more of the sheet comes into contact with a larger diameter roller, heating is even more important when using larger diameter rollers to prevent adhesion. While some drying is advantageous, drying the sheet too quickly during the calendering process could result in the introduction of fractures and other flaws within the structural matrix. A dryer sheet is less able to conform to a new shape without a rupture in the matrix than a wetter sheet subjected to the same level of shearing forces. Consequently, from this perspective the use of smaller diameter rollers is advantageous for reducing the drying effect of the reduction rollers.

In light of this, the diameter of the rollers should preferably be optimized and be sufficiently small to prevent overdrying of the material during the calendering process, while also being sufficiently large to reduce the amount of shearing force imparted to the sheet, thereby allowing a greater reduction of sheet thickness during each reduction step.

The optimization of the roller diameters in order to achieve the greatest amount of reduction of sheet thickness, while at the same time preventing overdrying of the hydraulically settable sheet, is preferred in order to reduce the number of reduction steps in a manufacturing process. Besides reducing the number of working parts, reducing the number of reduction steps also eliminates the number of rollers whose speed must be carefully synchronized in order to prevent sheet buildup behind the rollers (in the case of rollers rotating too slow) or sheet tearing (in the case of rollers rotating too fast).

Because each of the roller pairs reduces the thickness of the sheet as it passes therebetween, the sheet will speed up each time it passes through a set of rollers. Therefore, each of the roller pairs must independently rotate at the proper speed in order to prevent interruption of the calendering process. Eliminating the number of reduction steps greatly simplifies this synchronization problem.

The shearing force that is applied by the rollers on the sheets also corresponds to the speed of the rollers, including the differential between the sheet as it passes between a pair of rollers and the speed of the rollers. As the sheets are fed through the rollers at higher speeds, the amount of shear that is applied also increases. Therefore, the speed with which the hydraulically settable mixture is extruded and subsequently calendered can be optimized in order to reduce flaws caused by excess shear while maintaining the highest speed possible in order to maximize production of the sheets. The optimal speed with which a hydraulically settable mixture is first extruded and then calendered depends upon a number of interrelated variables including, for example, the rheology and other properties of the mixture, the thickness of the extruded sheet, the amount of reduction of the sheet, and the level of adhesion between the mixture and the rollers. In light of the various parameters, there is no preferred speed with which the sheet is extruded and then calendered.

As set forth above, it is preferable to treat the roller surfaces in order to prevent sticking or adhesion of the hydraulically settable sheet to the rollers. One method entails heating the rollers, which causes some of the water within the hydraulic mixture to evaporate and to create a steam barrier between the sheet and the rollers. Evaporation of some of the water also reduces the amount of water within the hydraulically settable mixture, thereby increasing the green strength of the sheet. The temperature of the rollers, however, must not be so high as to dry or harden the surface of the sheet to the point that would create residual stresses, fractures, flaking, or other deformities or irregularities in the sheet. Accordingly, it is preferable to heat the rollers to a temperature within the range from between about 50°C to about 140°C, more preferably to between about 70°C to about 120°C, and most preferably to between about 85°C to about 105°C.

In addition, the rate of drying of the sheet can be reduced by incorporating aggregates having a low specific surface area. Aggregates which have a greater specific surface area can more readily release any water absorbed within the aggregate compared to aggregates having a lower specific surface area.

Generally, the stickiness of the hydraulically settable mixture increases as the amount of water in the mixture is increased. Therefore, the rollers should generally be

heated to a higher temperature in cases where the mixture contains more water, which is advantageous because sheets containing a higher water content must generally have more of the water removed in order to obtain adequate green strength. Higher temperature rollers will increase the rate of water evaporation.

Finally, it has been found that heating the hydraulically settable mixtures of the present invention increases the rate of the hydration reaction between the hydraulic binder and water. It is known that concrete normally takes about 28 days to achieve its optimum strength. However, heating the hydraulic mixtures of the present invention makes it possible to obtain substantial hydration of the hydraulic binder in as little as one day. Because a substantial amount of the final strength can be obtained even before the hydration reaction has reached the standard 28 day level, heated hydraulically settable sheets of the present invention can achieve a substantial amount of their final strength within as little as 10 minutes.

Because heated rollers can drive off significant amounts of water and improve the form stability, the amount of acceptable sheet thickness reduction will generally decrease in each successive reduction step as the sheet becomes drier. This is because a drier, stiffer sheet can tolerate less shear before flaws are introduced into the structural matrix.

In an alternative embodiment, adhesion between the hydraulically settable sheets and rollers can be reduced by cooling the rollers to or below room temperature. Heating the mixture in the extruder to a relatively high temperature, for example, and then cooling the sheet surface causes the vaporizing water to condense, which is thought to create a thin film of water between the sheet and the roller. The rollers should be cool enough to prevent the surface of the sheet from adhering to the rollers, but not so cold to cause the sheet to freeze or become so stiff or inflexible that it will fracture or shatter during the calendering process.

Overcooling the material can also greatly retard the hydration reaction, although this may be desirable in some cases. Accordingly, it is preferable to cool the rollers to a temperature within the range from between about -20°C to about 40°C, more preferably to between about 0°C to about 35°C, and most preferably to between about 5°C to about 30°C.

In order to obtain the beneficial nonadhesive effects of cooling the rollers, it will generally be necessary to first heat the hydraulically settable mixture before or during the extrusion process to a temperature that is significantly higher than the temperature of the cooled rollers. This allows for the beneficial condensation of water vapor from the

heated mixture onto the cooled rollers, thereby creating a thin layer of lubricating water between the rollers and the hydraulically settable mixture. Accordingly, it will generally be preferable to heat the extruding mixture to a temperature within the range from between about 20°C to about 80°C.

Another way to reduce the level of adhesion between the rollers and the hydraulically settable sheet is to treat the roller surfaces in order to make them less amenable to adhesion. Rollers are typically made from polished stainless steel and coated with a nonstick material such as polished chrome, nickel, or teflon.

By varying the gap between the rollers, it is possible to cause the hydraulically settable sheet to spread or widen in the "X" direction from the point where the gap is more narrow toward the point where the gap is wider. Spreading or widening the sheet in the "X" direction also has the beneficial effect of reorienting some of the fibers in the "X" direction, thereby creating a sheet with bidirectionally oriented fibers (in the "X" and "Y" directions). Orienting the fibers maximizes the tensile strength imparting properties of the fibers in the direction of orientation.

In addition, orienting the fibers is particularly useful in order to reinforce a hinge or score within the sheet. Fibers which are greater in length than the width of the fold or bend can act as a bridge to connect the material on either side of the fold or bend even if the matrix is partially or even substantially fractured along the fold or bend. This bridging effect is enhanced if the fibers are generally aligned at an angle to the fold or bend.

Finally, it should be understood that due to the plastic nature and relatively high level of workability of the hydraulically settable mixture, the drying and finishing process will usually not result in much compression of the sheet. In other words, the density of the sheet will remain substantially the same throughout the calendering process, although some compaction would be expected, particularly where the sheet has been significantly dried while passing between other reduction rollers. Where compaction is desired, the sheet can be passed between a pair of compaction rollers 174, as shown in Figure 14, following a drying step as set forth more fully below.

One of ordinary skill in the art will appreciate that the extrusion step need not formally employ the use of an "extruder" as the term is used in the art. The purpose of the extrusion step is to provide a continuous, well-regulated supply of hydraulically settable material to the rollers. The extrusion step preferably orients the fibers in the direction of the flow of the material. This may be achieved by other mechanisms known to those skilled in the art to effect the "extrusion" or flow of material through an

appropriate opening. The force needed to cause a hydraulically settable mixture to flow may, for example, be supplied by gravity.

D. The Drying Process.

Although the calendering step often results in partial or even substantial drying of the hydraulically settable sheet, it will be preferable to further dry the sheet in order to obtain a sheet with the desired properties of tensile strength and toughness. This may be accomplished in a number of ways, each of which involves heating the sheet in order to drive off the excess water. A preferred method of drying the sheet involves the use of large diameter, heated drying rollers sometimes known in the art as "Yankee" rollers, although a series of smaller rollers may also be employed. The main concern is that the combined surface areas of the rollers be adequate to efficiently effectuate drying of the sheet.

In contrast to the reduction rollers, which are generally aligned in pairs of rollers, the drying rollers are individually aligned so that the sheet passes over the surface of each roller individually in sequence. In this way, the two sides of the hydraulically settable sheet are alternatively dried in steps. While the sheet passes between the reduction rollers during the calendering step in a generally linear path, the sheet follows a generally sinusoidal path when wrapping around and through the drying rollers during the drying step.

Figure 14 shows drying rollers 172, with the side of sheet 155 adjacent to the first drying roller heated by the first drying roller while the other side is exposed to the air. The heated sheet loses water in the form of vapor, which can escape out the sides of the roller or the surface of the sheet opposite the roller. The vapor also provides a nonstick barrier between the sheet and roller. The drying rollers may have tiny holes within the surface in order to allow some of the water vapor to escape through the holes during the drying step.

As sheet 155 continues on its path it is rolled onto a second drying roller where the other side of sheet 155 comes into contact with the roller surface and is dried (Figure 14). This process may be continued for as many steps as needed in order to dry the sheet in the desired amount. The amount of drying will depend on a number of factors, including the amount of water within the sheet, the thickness of the sheet, the time that the sheet is in contact with the roller surface, the temperature of the rollers, and the desired properties of the sheet. In some cases it may be preferable to dry one side of the sheet more than the other. This may be accomplished by designing a system which

maximizes the contact of one side of the sheet with the drying rollers while minimizing the contact of the other side.

The temperature of the drying rollers will depend on a number of factors, including the moisture content of the sheet as it passes over a particular roller. In any event, the temperature of the drying rollers should be less than about 300°C. Although the hydraulically settable material should not be heated above 250°C in order to prevent the destruction of the organic constituents (such as rheology-modifying agent or fibers), rollers heated to above this temperature may be used so long as there is adequate water within the mixture which can cool the material as the water vaporizes. Nevertheless, as the amount of water decreases during the drying process, the temperature of the rollers should be reduced to prevent overheating of the material.

In some cases, it may be preferable to use a drying tunnel or chamber in conjunction with the drying rollers. In order to obtain the full effect of heat convection drying, it is often preferable to circulate the heated air in order to speed up the drying process. The temperature within the drying tunnel, as well as the residence or dwell time of the sheet within the tunnel, will determine the amount and rate of evaporation of the water within the hydraulically settable mixture.

In order to achieve quick drying of the surface, it may be preferable to more quickly pass the sheet through a very hot drying tunnel. Conversely, in order to achieve a more uniform and deep drying of the sheet, it might be desirable to pass the sheet more slowly through the drying tunnel. The drying tunnel should not usually exceed 250°C in order to prevent the destruction of the cellulose fibers and rheology-modifying agents. In light of the foregoing, the drying tunnel will preferably be heated to a temperature within the range from between about 50°C to about 250°C, and more preferably within the range from between about 100°C to about 200°C.

In some cases, the drying process set forth above will be the final step before the sheet is either used to form a container or other object or, alternatively, rolled onto a spool or stacked as sheets until needed. In other cases, particularly where a sheet with a smoother, more paper-like finish is desired, this drying step will be followed by one or more additional steps set forth more fully below, including a compacting step and/or a finishing step. In the case of compaction, it is generally preferable to leave the sheets with some amount of moisture to prevent fracturing of the matrix during the optional compaction step. Otherwise, if the drying step is not followed by a compaction step, it is generally desired to substantially dry out the sheet in order to quickly maximize the tensile strength and toughness of the sheet.

E. Optional Finishing Processes.

In many cases, it may be desirable to calender the hydraulically settable sheet in order to achieve the final thickness, tolerance, and surface finish. In addition, the compaction process can be used to remove unwanted voids within the structural matrix. Referring to Figure 14, sheet 155 may be optionally passed between a pair of calender rollers 174 after being substantially dried by drying rollers 172. The compaction process generally yields a sheet with higher density and strength, fewer surface defects, and a reduced thickness. The amount of compressive force of the compaction rollers can be adjusted to correspond to the particular properties of the sheet.

The compaction process preferably yields a sheet of reduced thickness and increased density without causing further elongation of the sheet and without negatively disrupting or weakening the structural matrix. In order to achieve compaction without elongating the sheet and without weakening the structural matrix, it is important to control the drying process so that the sheet contains an amount of water within an optimum range. If the sheet contains too much water, the compaction rollers will elongate the sheet in a similar fashion as the forming rollers. In fact, the compaction rollers are substantially the same as the forming rollers, the only difference being that compaction, rather than elongation will occur if the sheet is dry enough.

On the other hand, overdrying the sheet prior to the compaction step can yield a weaker sheet. At some point the hydraulically settable sheet can become so brittle and rigid that the structural matrix cannot be compressed without fracturing. The fracturing of the structural matrix can diminish the final strength of the sheet even if the fractures are microscopic and not visible to the naked eye. Nevertheless, the compaction process of a dry sheet may be improved by spraying the surface of the sheet with water, which provides the sheet with adequate moisture and also fixes and aligns the compacted particles within the sheet surface.

Because the compaction process (including one or more compaction steps) usually involves a slightly moist sheet, it is usually preferable after the compaction step to further dry the sheet in a manner similar to the drying process outlined above using optional drying rollers 178 as shown in Figure 14. This optional drying step may be carried out using drying rollers, a drying tunnel, or a combination of the two. Nevertheless, in some cases the sheet may be further processed without a second drying step, such as where the sheet is immediately used to form a container or other object, is scored, or where it is otherwise advantageous to have a slightly moist sheet.

It may also be preferable to further alter the surface of the hydraulically settable sheet by passing the sheet between one or more pairs of calendering rollers 180 as shown in Figure 14. For example, in order to create a sheet with a very smooth surface on one or both sides, the sheet may be passed between a pair of hard and soft rollers. The term "hard roller" refers to a roller having a very polished surface and which leaves the side of the sheet in contact with the hard roller very smooth. The term "soft roller" refers to a roller having a surface capable of creating enough friction between the soft roller and the sheet that it pulls the sheet through the hard and soft roller pair. This is necessary because the hard roller is usually too slick to pull the dry sheet through a pair of hard rollers. Besides, some slippage of the hard roller is advantageous in order to align the particles on the surface of the sheet. The finishing process may be optionally facilitated by spraying water on the sheet surface, and/or by coating the surface with clay, calcium carbonate, or other appropriate coating materials known to one of ordinary skill in the art.

In other embodiments, calendering rollers 180 can impart a desired texture such as a meshed or checkered surface. Instead of using a hard and a soft roller, rollers which can imprint the sheets with the desired finish may be used. If desired, the rollers can imprint the surface of the sheet with a logo or other design. Special rollers capable of imparting a water mark can be used alone or in conjunction with any of these other rollers.

F. <u>Coating Processes</u>.

It may be preferable to coat the hydraulically settable sheet prepared using the processes set forth above. Coatings can be used to alter the surface characteristics of the hydraulic sheet in a number of ways. They may provide protection against moisture, base, acid, or oil-based solvents. They may also provide a smoother or glossier surface. They may even reinforce the hydraulically settable sheet, particularly at a bend or fold line. As set forth above, some coatings can be applied to the surface of the sheet during the sheet forming process, in which case the process is an "on-machine" process. However, it may be preferable to apply the coating after the sheet forming process, in which case the process is an "off-machine" process.

The object of the coating process is usually to achieve a uniform film with minimum defects on the surface of the sheet. The selection of a particular coating process depends on a number of substrate (*i.e.*, sheet) variables, as well as coating formulation variables. The substrate variables include the strength, wettability, porosity, density, smoothness, and uniformity of the sheet. The coating formulation variables

include total solids content, solvent base (including water solubility and volatility), surface tension, and rheology. Coating processes known in the art that may be used to coat the hydraulically settable sheets of the present invention include blade, puddle, air-knife, printing, and gravure coating. In other cases, it may be preferable to spray the coating onto the surface of the sheet using spraying means known in the art.

G. Other Processes.

It may be desirable to apply print or other indicia on the surface of the sheet such as a watermark, company identification mark, logo, or trademark. This can be accomplished using printing means known in the art of printing paper or cardboard products. Because the sheets have a relatively high porosity like paper or cardboard, the applied ink will tend to dry rapidly. In addition, decals, labels or other indicia can be attached or adhered to the hydraulically settable sheet using methods known in the art.

Finally, the substantially hardened sheets can be immediately used to form containers, printed materials, or other objects, or they may be stored until needed such as, for example, by winding the sheets into a spooler 182 as shown in Figure 14, or cutting and stacking individual sheets onto a pile.

The hydraulically settable sheets made according to the processes set forth above can then be used just like paper or cardboard and can be fashioned into an endless variety of containers or other useful objects. One particularly valuable use of the sheets of the present invention is in the manufacture of disposable food or beverage containers used in the fast food industry. Such containers can be manufactured at a fraction of the cost of comparable containers made from paper or polystyrene. In addition, such containers are far more environmentally friendly because they readily decompose into mainly common inorganic substances found naturally within the earth. The organic components such as fibers or rheology-modifying agents are readily biodegradable and, in any event, comprise only a fraction of the overall mass of the containers made from the sheets used in the present invention.

VI. EXAMPLES OF THE PREFERRED EMBODIMENTS.

The following examples disclose the preparation of various hydraulically settable compositions useful in forming the hinges of the invention.

Examples 1-4

Hydraulically settable sheets were formed from the following cementitious mixtures containing hollow glass spheres (diameter <100 microns) as the aggregate in order to form lightweight plate-like objects.

Example	Cement	Water	Tylose® FL 15002	Glass Spheres
1	4 kg	2.18 kg	200 g	445 g
2	3 kg	1.85 kg	150 g	572 g
3	2 kg	1.57 kg	100 g	857 g
4	1 kg	1.55 kg	100 g	905 g

The cementitious mixtures were prepared by first combining the hydraulic cement, Tylose®, and water together using a high shear mixer for 5 minutes. Thereafter, the glass spheres were added and mixed for 5 minutes using a low shear mixer. The resultant cementitious mixtures in Examples 1-4 had water to cement ratios of approximately 0.55, 0.62, 0.79, and 1.58, respectively. Even with the high water to cement ratio of Example 4, the cementitious mixture was form stable in the green state and readily moldable. The percentage by weight of the glass spheres in each of Examples 1-4 was 6.5%, 10.3%, 18.9%, and 25.3%, respectively.

The cementitious mixtures were formed into relatively thick sheets having a thickness ranging from 2.0-4.0 mm. The resulting sheets were extremely lightweight, having densities in the range from about 0.25 to 0.5. Tests showed that these extremely lightweight materials were highly insulating. In fact, 2.0 mm thick sheets were placed in an oven at 150°C for three hours; thereafter, they could be removed by hand. This means that the surface temperature was significantly less than 60°C, which may be due to the relatively low specific heat of the lightweight hydraulically settable materials made in these examples.

The sheets of Examples 1-4 were score cut when dried using a knife blade cutter to form hinges therein. The score cut had a triangular profile and resulted in the material below the score forming the hinges having a thickness of 0.1 mm.

Examples 5-8

The cementitious mixtures of Examples 1-4 were altered by adding varying amounts of abaca fiber, which were blended in during the high shear mixing step.

Example	Corresponding Example	Amount of Abaca fiber
5	1	149 g
6	2	152 g
7	3	180 g
8	4	181 g

The resultant percentage by weight of the abaca fibers in Examples 5-8 was 2.1%, 2.7%, 3.8%, and 4.8%, respectively. The hydraulically settable sheets formed therefrom were as lightweight and insulative as those made in Examples 1-4, but were much tougher and had a higher fracture energy. In addition, adding more fibers made the sheets somewhat more flexible, which would make them more useful in, *e.g.*, containers having hinged flaps or other closure mechanisms. Hence, this example demonstrated that the use of abaca fibers, as well as other types of fibers, allowed the formation of hydraulically settable sheets having more flexibility, ductility, and toughness than what was previously thought possible.

The sheets of Examples 5-8 were score cut when dried using a continuous die cut roller to form hinges therein. The score cut was made at a 45° angle to the direction of the fibers in the sheets. The score cut had a triangular profile and resulted in the material below the score forming the hinges having a thickness of 0.05 mm.

Examples 9-11

Hydraulically settable sheets were prepared using the mix design and procedures set forth in Example 1 (*i.e.*, 4 kg of portland white cement), with the exception that aluminum powder (<100 mesh) and NaOH were added to the cementitious mixtures in the following amounts:

Example	Aluminum	NaOH
9	4 g	21.9 g
10	6 g	34.7 g
11	8 g	34.7 g

The NaOH was added to the cementitious mixture to activate the aluminum by establishing a pH in the preferable range of about 13.1-13.8. In addition, the molded plate-like sheets were heated to about 80°C for 30-60 minutes. This resulted in a final hardened material with increased porosity, decreased bulk density, and higher insulation ability. It was also shown that the rate and extent of the reaction of aluminum metal can be altered by adjusting the amount of aluminum and heat that are added. As more of each is added, the material becomes lighter, fluffier and softer, making good cushioning material.

It is important to note that shrinkage cracks were not observed in the sheets of Examples 9-11, even though the cementitious mixtures were heated and much of the water was driven off rapidly.

The sheets of Examples 9-11 had double score cuts made therein to form hinges that allowed bending of the sheets in either direction. The score cuts had a triangular profile and were made on both sides of the sheets at adjacent locations.

Examples 12-14

Hydraulically settable sheets were formed from cementitious mixtures containing 4 kg of portland white cement, 1.4 kg of water, and 40 g of Tylose® FL 15002, along with the following amounts of aluminum, NaOH, and abaca fibers:

Example	Aluminum	NaOH	Abaca Fibers
12	10.0 g	22.3 g	60 g
13	15.0 g	22.3 g	60 g
14	22.5 g	22.3 g	60 g

The cementitious mixtures were prepared substantially according to the procedures set forth in Example 1, with the exception that fibers rather than hollow glass spheres added. Like the cementitious mixtures of Examples 9-11, these materials were extremely lightweight and very insulative because of the amount of air that was incorporated into the hydraulically settable mixtures. The hydraulically settable sheets of these examples have increased toughness and fracture energy because of the addition of the fibers.

The sheets of Examples 12-14 were pressed while wet with a scoring die and flexed to form living hinges therein. The pressed scores had a rounded profile and were made at a 35° angle to the direction of the fibers in the sheets. The score was pressed at

a controlled rate, depth and pressure such that the material below the score forming the hinges had a thickness of 0.1 mm.

Examples 15-18

Hydraulically settable sheets were formed from cementitious mixtures containing 4 kg of portland white cement, 1.96 kg of water, 200 g of Tylose® FL 15002, and 60 g of abaca fiber, along with the following amounts of aluminum, NaOH, and hollow glass spheres:

	Glass Spheres				
Example	Fine	Medium	Coarse	Aluminum	NaOH
15	133 g	317 g	207 g	4.0 g	19.7 g
16	133 g	317 g	207 g	6.0 g	31.2 g
17	133 g	317 g	207 g	8.0 g	31.2 g
18	133 g	317 g	207 g	0.0 g	0 g

The cementitious mixtures were prepared substantially according to the procedures set forth in Examples 9-11, with the exception that hollow glass spheres having three different diameters were used. The average diameter of the hollow glass spheres designated as "fine" was 30 microns; of the "medium" was 47 microns; and of the "coarse" was 67 microns. The percentage by weight of the total amount of glass spheres in each of the cementitious mixtures of Examples 15-17 was 2.1%. Of course, Example 18 does not incorporate aluminum and NaOH.

The hydraulically settable sheets formed therefrom were relatively thick, (2.5 mm), were extremely lightweight (specific gravity < 0.7) and were very insulative because of the amount of air and the effective packing of the glass balls incorporated into the mixtures. The cementitious mixtures of these examples demonstrated the value of packing the aggregates in order to maximize their effect in the resultant composition. While the cementitious mixture of Example 18 is a good composition for many circumstances, its insulative capabilities are not as great as the cementitious mixtures of Examples 15-17.

The sheets of Examples 15-18 were pressed while wet at a controlled rate, depth, and pressure with a scoring die on both sides thereof in order to form a double score hinge. The pressed double scores were formed at a 60° angle to the direction of the fibers

and had a rectangular profile. The formed hinges allowed bending of the sheets in either direction.

Examples 19-20

Cementitious mixtures containing the following components were used to make hydraulically settable sheets:

Example	Cement	Water	Tylose® FL 15002	Abaca Fibers	Surfactant
19	10 kg	23.0 kg	300 g	200 g	300 g
20	10 kg	20.0 kg	300 g	200 g	300 g

In these examples, microfine cement was utilized to make the hydraulically settable sheets. The cementitious mixtures were made by mixing the components for about 10 minutes in a high energy mixer of the type discussed above, which is available from E. Khashoggi Industries. This high energy and high speed mixer introduced significant amounts of air into the cementitious mixtures. The air was entrained within the cementitious mixture by use of the surfactant and stabilized by the Tylose®. The resulting cementitious mixtures were passed between a pair of rollers and formed into relatively thin sheets (1 mm). The sheets were more quickly dried by passing them through a heat tunnel. These sheets had a specific gravity of between 0.25 and 0.4.

The sheets were subsequently scored, folded into the shape of a cereal box, and glued together using adhesive techniques known in the art. From these examples it was learned that scoring relatively thin hydraulically settable sheets allowed the sheets to be folded or bent much like paper or cardboard products of the same general thickness.

Examples 21-22

Cementitious mixtures containing the following components were used to make hydraulically settable sheets:

Example	Cement	Water	Tylose® FL 15002	Graphite Fibers	Surfactant
21	4.23 kg	8.1 kg	120 g	260 g	135 g
22	10.0 kg	20.0 kg	300 g	300 g	300 g

In these examples, microfine cement was utilized. Like the products of Examples 19 and 20, the hydraulically settable mixtures of these examples were made by mixing the components for about 10 minutes in a high shear mixer of the type discussed above. This high shear mixer introduced significant amounts of air into the cementitious mixtures and the air was entrained within the cementitious mixture by the surfactant.

However, due to the graphite fibers, the mixture was not as easily foamed and was not as lightweight and insulative as materials containing no graphite fibers. The resulting cementitious mixtures were passed between a pair of rollers and formed into relatively thin sheets (1 mm), which were subsequently scored, folded into the shape of a cereal box, and glued together using adhesive techniques known in the art.

Example 23

Relatively thin hydraulically settable sheets were formed by molding a cementitious mixture which included the following:

Portland White Cement	1.0 kg
Water	2.5 kg
Tylose® FL 15002	200 g
Hollow Glass Spheres (<100 microns)	1.0 kg
Abaca Fiber	10% by volume

The cementitious mixture was made by prewetting the abaca fiber (which was pretreated by the manufacturer so that greater than 85% of the cellulose is α-hydroxycellulose) and then adding the excess water and the fibers to a mixture of Tylose® and portland cement. This mixture was mixed at relatively high speed for about 10 minutes, and then at a relatively slow speed for 10 minutes after the hollow glass spheres were added. The resulting cementitious mixture had a water to cement ratio of approximately 2.5.

This mixture was passed between a pair of rollers and formed into relatively thin sheets having a thickness of about 1 mm. Wet sheets were scored and then folded in an attempt to create a box. There was, however, a fair amount of splitting and a box with sufficient strength and integrity could not be formed.

Thereafter, sheets were first allowed to harden and then were scored, folded into the shape of a box, and glued together by cementing or gluing methods well-known in

the paper art. The amount of splitting at the fold was negligible, which demonstrated that it is preferable to score and then fold the thin sheets after they have been allowed to harden or solidify somewhat. The thin sheets were formed into a box that had the shape, look and weight of a dry cereal box manufactured from cardboard stock. Example 23 demonstrates that it is possible to use cementitious materials to make boxes or other containers of similar shape that are presently made from cardboard, paper, or plastic.

The following examples demonstrate that highly flexible hydraulically settable sheets having high toughness and strength can be manufactured. They are useful in containment applications where cushioning and flexibility are of particular interest.

Examples 24-28

Flexible, cushioning sheets were formed from cementitious mixtures containing the following:

Example	Plastic Spheres	Cement	Water	Tylose®
24	0.12 kg	1.0 kg	2.0 kg	0.1 kg
25	0.1213 kg	0.8 kg	2.0 kg	0.1 kg
26	0.1225 kg	0.6 kg	2.0 kg	0.1 kg
27	0.1238 kg	0.4 kg	2.0 kg	0.1 kg
28	0.1251 kg	0.2 kg	2.0 kg	0.1 kg

The "plastic spheres" are made from polypropylene and have average particle sizes less than 100 microns and an average density of 0.02 g/cm^3. The cementitious mixtures were mixed and then formed into sheets according to the procedure set forth in Example 23. The hydraulically settable sheets were relatively strong and very flexible compared to previous mix designs. The compressive strength of the plate made according to Example 24 was 2 MPa and the tensile strength was 1 MPa. The surprising feature is that the compressive and tensile strengths are of the same magnitude, which is very unusual for most cement products. Usually the compressive strength is far greater than tensile strength. As less cement is added, the compressive and tensile strengths decrease in increments, with the plate of Example 28 having a tensile strength of 0.5 MPa.

These packaging materials could be physically compressed without crumbling even when subject to forces that were greater than forces normally experienced by polystyrene containment materials. The flexible hydraulically settable materials were

alternatively extruded into the shape of rectangular shaped bars, which more dramatically demonstrated the degree of flexibility made possible by this mixture.

The densities of the hydraulically settable packaging materials made in these examples ranged between 0.1 and 0.6 g/cm^3, with the density decreasing as less cement is used.

The sheets of Examples 24-28 were pressed in the wet state with multiple scores having a rounded profile on one side of the sheet to form a hinge therein. These multiple score hinges provided increased bending of the sheets compared to sheets with a single score hinge.

Examples 29-33

Flexible hydraulically settable sheets were made according to Examples 24-28, except that prewetted abaca fibers were added to the cementitious mixture in the following amounts, as measured by unit volume:

Example	Abaca Fiber
29	2%
30	4%
31	6%
32	8%
33	10%

The fibers were well-dispersed throughout the hydraulically settable mixture using a high shear mixer. The resulting hydraulically settable sheets made therefrom had substantially the same densities and flexibilities as those in Examples 24-28, but with increasing tensile strengths as the amount of abaca fiber was increased. The tensile strengths of the materials formed herein ranged up to 5 MPa.

The sheets of Examples 29-33 were pressed in the wet state with multiple scores having a rectangular profile on one side of the sheet to form a hinge therein. The scores were pressed into the sheets at a 20° angle from the direction of the fibers in the sheets. The hinges allow the sheets to be formed into a variety of containers.

Example 34

Hydraulically settable containers are formed using any of the compositions and procedures set forth in Examples 24-33, except that the plastic balls are concentrated near

the surface of the cementitious mixture, yielding a molded material in which the plastic balls are concentrated at or near the surfaces of the final hardened product. The sheets and containers formed therefrom have a higher concentration of plastic balls near the surface of the cement matrix where flexibility is more important, and virtually no plastic balls in the center of the objects where flexibility is less important. The advantage of this is greater flexibility at the surfaces with the same amounts or less of plastic balls in the overall compositions. At the same time, the rigidity of the center of the container walls makes them as durable and tough as the more rigid containers above.

Example 35

Using any of the foregoing compositions, a hydraulically settable sheet is molded, scored and then fashioned into the shape of a carton. Depending on the composition, the carton will exhibit high strength, durability, flexibility, low weight, and/or low density.

Example 36

Using any of the foregoing compositions, a hydraulically settable sheet is molded and then fashioned into the shape of a box. This may be carried out by extrusion, and/or calendering, and/or score cutting, and/or folding. Depending on the composition, the box will exhibit high strength, durability, flexibility, low weight, and/or low density.

In the following examples, very thin sheets were formed (0.1-0.5 mm) having characteristics and properties that made them suitable for use much like paper, cardboard, plastic, polystyrene, or metal sheets of similar thickness and weight. The desired properties were designed into the sheets using a microstructural engineering approach. This allowed for the manufacture of sheets having a variety of desirable properties, including properties not generally possible using mass-produced sheet-like objects presently manufactured from the foregoing materials.

Examples 37-52

Sheets capable of being formed into a variety of objects (including food and beverage containers) were manufactured from a moldable cementitious mixture that contained the following components:

Portland Cement	1.0 kg
Perlite	0.5 kg
Mica	0.5 kg
Fiber (Southern pine)	0.25 kg
Tylose® FL 15002	0.2 kg
Water	2.5 kg

The portland cement, mica, fiber, Tylose®, and water were mixed together in a high shear mixer for 5 minutes, after which the perlite was added and the resulting mixture mixed for an additional 5 minutes in a low shear mixer. The cementitious mixture was then placed into an auger extruder and extruded through a die having an opening in the shape of a slit. Continuous sheets having a width of 300 mm and a thickness of 6 mm were extruded.

The sheets were thereafter passed between one or more pairs of reduction rollers in order to obtain sheets having final thicknesses of 0.2 mm, 0.25 mm, 0.3 mm, 0.35 mm, 0.4 mm, 0.45 mm, and 0.5 mm, respectively. The rollers had a diameter of 17 cm and were made of stainless steel coated with polished nickel to aid in preventing the cementitious mixture from sticking to the rollers. In addition, the rollers were heated to a temperature of 110°C to further prevent sticking between the mixture and the rollers.

In order to obtain sheets having the desired thickness, the extruded sheets were reduced in steps by using reduction roller pairs having progressively smaller gap distances between the rollers. The sheet thicknesses were reduced as follows:

6 mm ==> 2 mm ==> 0.5 mm ==> final thickness (0.45 mm, 0.4 mm, 0.35 mm, 0.3 mm, 0.25 mm, or 0.2 mm)

A combination of the extrusion process and the calendering process yielded sheets with substantially unidirectionally oriented fibers along the length (or direction of elongation) of the sheet. Because of this, the sheets had higher tensile strength in the lengthwise direction (10-12 MPa) compared to the widthwise direction (5-6 MPa).

The hardened hydraulically settable sheets were finished, coated, and then formed into a number of different food and beverage containers.

A "clamshell" container (such as those presently used in the fast food industry to package hamburgers) was made by cutting an appropriate shape from a sheet, score cutting the sheet to form the desired fold lines, folding the sheet into the shape of a clamshell container, and adhering the ends of the folded sheet (using both adhesive and

interlocking flap means) to preserve the integrity of the container. Sheets having thicknesses of 0.4 mm and 0.5 mm were used to make the clamshell containers.

The sheet was found to more easily bend or close together on the side of the sheet opposite the score cut. It should be noted that normal scores in conventional materials generally allow the sheet to more easily bend or close together on the side of the score. The resulting clamshell containers exhibited comparable or superior insulating ability compared to paper clamshells.

A french fry container (such as those used to serve cooked french fries in the fast food industry) was made by cutting an appropriate shape from a sheet, score cutting the sheet to form the desired fold lines, folding the sheet into the shape of a french fry container, and adhering the ends of the folded sheet using adhesive means to preserve the integrity of the container. Sheets having thicknesses of 0.25 mm, 0.3 mm, 0.35 mm, 0.4 mm, 0.45 mm, and 0.5 mm were used to make the french fry containers.

A frozen food box (such as those used by supermarkets to package frozen foods such as vegetables or french fries) was made by cutting an appropriate shape from a sheet, score cutting the sheet to form the desired fold lines, folding the sheet into the shape of a frozen food box, and adhering the ends of the folded sheet using adhesive means to preserve the integrity of the box. Sheets having thicknesses of 0.25 mm, 0.3 mm, 0.35 mm, 0.4 mm, 0.45 mm, and 0.5 mm were used to make the frozen food boxes.

A cold cereal box was made by cutting an appropriate shape from a 0.3 mm thick sheet, score cutting the sheet to form the desired fold lines, folding the sheet into the shape of a cold cereal box, and adhering the ends of the folded sheet using adhesive means to preserve the integrity of the cereal box.

A straw was made by rolling a piece of a 0.25 mm sheet into the form of a straw and adhering the ends together using adhesion means known in the art. A hinge was molded in one section of the straw to make the straw bendable. In making the straw, as in making each of the containers set forth above, it was advantageous to remoisten the sheet somewhat in order to temporarily introduce a higher level of flexibility into the sheet. This minimized splitting and tearing of the sheet.

The containers were found to break down in the presence of water over time, with one day being the average time of disintegration. The excess waste material that was trimmed from the sheets when making the containers was easily recycled by simply breaking it up and mixing it back into the hydraulically settable mixture.

The various containers that were made are set forth as follows, including the thickness of the sheet used to make each container:

Example	Container	Sheet Thickness
37	clam shell	0.4 mm
38	clam shell	0.5 mm
39	french fry box	0.25 mm
40	french fry box	0.3 mm
41	french fry box	0.35 mm
42	french fry box	0.4 mm
43	french fry box	0.45 mm
44	french fry box	0.5 mm
45	frozen food box	0.25 mm
46	frozen food box	0.3 mm
47	frozen food box	0.35 mm
48	frozen food box	0.4 mm
49	frozen food box	0.45 mm
50	frozen food box	0.5 mm
51	cold cereal box	0.3 mm
52	drinking straw	0.25 mm

Example 53

The hydraulically settable sheets used to manufacture the containers in Examples 37-52 were printed using conventional printing presses used to print conventional paper sheets. The ink was able to dry as fast or faster than conventional paper sheets. The printed sheets could then be formed into any of the containers listed in Examples 37-52 above.

Example 54

Clamshell containers were made using the sheets made according to Examples 37-52. The sheets were tested to determine the optimum score cut depth which would allow for the easiest bend, while at the same time leaving a hinge with the highest strength and resilience. Score depths ranging between 20% to 50% were tested, with a score depth of 25% yielding the best results. In addition, it was found that thicker sheets (0.4-0.5 mm) gave a better score and yielded a stronger, more rigid clamshell container.

Example 55

A clamshell was made using the sheets of Examples 37-52, except that a triple reverse hinge was used. That is, a series of three score cuts were cut into the outer side of the claim shell container. Because this decreased the distance that each individual score line had to bend, the resulting hinge could be opened and closed more times without breaking compared to a single score cut hinge.

Example 56

Clamshell containers made according to Examples 37 and 38 were passed through a commercial wax coating machine, whereby a uniform layer of wax was applied to the surface. The layer of wax completely sealed the surface of the cup to moisture and rendered it watertight.

Example 57

Clamshell containers made according to Examples 37 and 38 were coated with an acrylic coating using a fine spraying nozzle. As did the wax in Example 56, the layer of acrylic coating completely sealed the surface of the cup to moisture and rendered it watertight. However, the acrylic coating had the advantage that it was not as visible as the wax coating. Because a thinner acrylic coating was possible, the cup looked almost as if it were uncoated. The glossiness of the cup could be controlled by using different types of acrylic coatings.

Example 58

Clamshell containers made according to Examples 37 and 38 were coated with a commercially used melamine coating using a fine spraying nozzle. As in Examples 56 and 57, the layer of melamine coating completely sealed the surface of the cup to moisture and rendered it watertight. However, the melamine coating was also less visible and could be applied in a thinner coat compared to the wax coating. The glossiness of the cup could be controlled by using different types of melamine coatings.

Example 59

Clamshell containers made according to Examples 37 and 38 were coated with a totally environmentally sound coating consisting of a mixture of hydroxymethylcellulose plasticized with polyethylene glycol. This coating completely sealed the surface of the cup to moisture and rendered it watertight. However, the surface

looked even more natural and less glossy compared to cups coated with wax, acrylic, or melamine.

Examples 60-63

French fry containers made according to Examples 39-44 were alternatively coated with the same coating materials used to coat the clamshell containers in Examples 56-59. The results were substantially identical to those achieved with the coated clamshell containers.

Example	Coating Material
60	wax
61	acrylic
62	melamine
63	plasticized hydroxymethylcellulose

Examples 64-67

Frozen food containers made according to Examples 45-50 were alternatively coated with the same coating materials used to coat the clamshell containers in Examples 56-59. The results were substantially identical to those achieved with the coated clamshell containers.

Example	Coating Material
64	wax
65	acrylic
66	melamine
67	plasticized hydroxymethylcellulose

Examples 68-71

Cold cereal boxes made according to Example 51 were alternatively coated with the same coating materials used to coat the clamshell containers in Examples 56-59. The results were substantially identical to those achieved with the coated clamshell containers.

Example	Coating Material
68	wax
69	acrylic
70	melamine
71	plasticized hydroxymethylcellulose

Examples 72-75

Drinking straws made according to Example 52 are alternatively coated with the same coating materials used to coat the clamshell containers in Examples 56-59. The results are substantially identical to those achieved with the coated clamshell containers with regard to the outer surface of the straws, although it is more difficult to adequately coat the inside of the straw in this manner.

Example	Coating Material
72	wax
73	acrylic
74	melamine
75	plasticized hydroxymethylcellulose

Example 76

Containers set forth above were placed in a microwave oven and tested for microwave compatibility. The containers were tested to determine whether the containers themselves, or the food items within them, become hot when container and food were exposed to microwave radiation. Although the containers may have been expected to absorb some of the radiation and become hot in light of the water tied up within the hydraulically settable structural matrix, in fact, the containers themselves remained cool. Because of the low dielectric constant of the material, all of the energy was found to go into the food and not the container.

For the same reason, steam, which may have condensed onto the surface of the container during initial stages of the microwaving, was found to quickly revaporize under further microwaving. Therefore, when the food container was opened, no condensed steam was found on the surface of the container after the microwave process. Any excess steam comes out when the container is opened, leaving food which looks and tastes better. This is in sharp contrast to polystyrene containers which tend to accumulate large amounts of condensed steam on the container surfaces, thereby rendering a "soggier," and

hence less desirable, food product. In addition, polystyrene containers often melt if the food is heated too long.

The specific heats of the hydraulically settable materials of the present invention are relatively low, being about 0.9 J/g·K and having a low thermal constant within the range of 0.06-014 W/m·K. This allows for less thermal conductance from the food to the container during the microwave process. It was possible, therefore, to remove the container from the microwave without burning the hands. After the container was removed from the microwave oven it slowly warmed (by absorbing some of the heat within the food) but never become too hot to touch.

Example 77

Flat paper sheets suitable for manufacturing a wide variety of food and beverage containers were manufactured from a hydraulically settable mixture containing the following:

Portland Cement	1.0 kg
Perlite	0.3 kg
Hollow Glass Spheres (< 0.1 mm)	0.8 kg
Mica	0.5 kg
Fiber (Southern pine)	0.25 kg
Tylose® FL 15002	0.2 kg
Water	2.6 kg

The cement, mica, fiber, Tylose®, and water were mixed together in a high shear mixer for 5 minutes, after which the perlite and hollow glass spheres were added and the resulting mixture mixed using low shear. The mixture was extruded using an auger extruder and a die into a sheet 30 cm wide and 0.6 cm thick. The sheet was passed successively between pairs of heated rollers in order to reduce the thickness of the sheet to between 0.1 mm and 2 mm.

As a result of the lower specific surface area of the glass spheres (200-250 m^2/kg) compared to perlite, the mixture of Example 77 yielded a product with a more uniform thickness and improved surface finish compared to the mix design of Examples 37-52. The reduced specific surface area of the aggregates reduced the amount of moisture that was removed when contacting the heated calendering rollers. The material, therefore,

remains more moldable, retains the optimum rheology, and results in less microdefects and more uniformity during the calendering process.

Example 78

The mix design and molding processes of Examples 37-52 were repeated in every way except that the mica was substituted with 0.5 kg kaolin. The sheets made using this alternative mix design yielded sheets that had a glossier surface than where mica was used. The glossier surface resulted from the alignment of the smaller kaolin particles within the sheet surface when the sheet was successively passed between a pair of calendering rollers, which also acted like a pair of smoothing rollers.

Example 79

The mix design and molding process of Example 78 were repeated in every way except that 1.0 kg of kaolin was used. The sheets that were molded using this increased amount of kaolin had a smoother surface finish than when only 0.5 kg kaolin was used.

Example 80

The mix design and molding process of Example 78 were repeated in every way except that 1.5 kg of kaolin was used. The sheets that were molded using this increased amount of kaolin had a smoother surface finish than when only 0.5 kg or 1.0 kg of kaolin was used. However, the increase in kaolin yield a more brittle sheet. In addition, drying defects due to the increased specific surface area were somewhat problematic when passing the sheet between the reduction rollers.

Example 81

The mix design and molding processes of Examples 37-52 were repeated in every way except that the perlite was excluded and the amount of mica was increased to 1.5 kg. The resulting sheets made using this alternative mix design had a smoother finish. However, the hydraulically settable structural matrix was more dense and more brittle. In addition, there was an increase in drying defects.

Example 82

The mix design and molding process of Examples 37-52 were repeated in every way except that the amount of perlite was increased to 1.0 kg. The resulting sheets and

containers made therefrom had a slightly lower density with slightly lower strength and toughness.

Example 83

The mix design and molding process of Examples 37-52 were repeated in every way except that the amount of perlite was increased to 0.75 kg. The resulting sheets and containers made therefrom had a slightly lower density with slightly lower strength and toughness. However, the strength characteristics were somewhat better than when 1.0 kg of perlite was used, as in Example 82.

Example 84

The mix design and molding process of Examples 37-52 were repeated in every way except that the amount of perlite was reduced to 0.25 kg. The resulting sheets and containers made therefrom had a higher fiber content, slightly higher density, and greater strength and toughness.

Example 85

The mix design and molding process of Examples 37-52 were repeated in every way except that perlite was eliminated from the mix design altogether. The resulting sheets and containers made therefrom had a slightly higher density with greater strength and toughness.

The following examples relate to tests that were performed in order to optimize the mix designs that would yield products having the preferred performance criteria. Although only sheets were made in these examples, it will be understood to one of ordinary skill in the art how such sheets could be formed into appropriate food or beverage containers using any of the methods (including the examples) set forth within the specification relating to forming hinges in hydraulically settable sheets. In addition, many of the mix designs could also have application in either direct molding or wet sheet molding of food or beverage containers.

Examples 86-91

Hydraulically settable sheets having a thickness of 0.4 mm were manufactured according to the processes set forth in Examples 37-52 from a hydraulically settable mixture containing the following components:

Portland Cement	1.0 kg
Perlite	variable
Mica	0.5 kg
Tylose® FL 15002	0.2 kg
Fiber (Southern pine)	0.25 kg
Water	variable

The effect of adding varying amounts of perlite was studied to determine the effect on the properties of the material, particularly the strength properties of the hardened sheet. Because of the water-absorbing behavior of perlite, it was necessary to decrease the amount of water as the amount of perlite was decreased in order to maintain the same level of rheology and workability.

Example	Perlite	Water
86	0.5 kg	2.15 kg
87	0.4 kg	2.05 kg
88	0.3 kg	1.85 kg
89	0.2 kg	1.65 kg
90	0.1 kg	1.50 kg
91	0.0 kg	1.40 kg

The extrusion and calendering processes had the effect of longitudinally orienting the fibers in a substantially unidirectional manner. Therefore, the sheets possessed a "strong" and a "weak" direction. The sheets were tested for tensile strength in the two directions, designated as 0° for the strong direction and 90° for the weak direction. The level of elongation before failure and Young's modulus of elasticity were also tested.

The sheets were also tested for strength in the intermediate, or 45°, direction although only exemplary results for tests in this direction are given. The tensile strength, elongation, and Young's modulus of the sheets in the 45° direction generally fell between those measured in the strong and weak directions, although as a general rule they were closer to the same properties measured in the weak direction. The results are set forth as follows:

Example	Strength (MPa) 0°	Strength (MPa) 90°	Elongation (ΔL/L) 0°	Elongation (ΔL/L) 90°	Modulus (MPa) 0°	Modulus (MPa) 90°
86	10.67	5.18	1.57%	0.66%	2297	1375
87	11.2	5.33	2.38%	1.25%	2156	1559
88	13.4	6.27	2.22%	1.00%	2956	1548
89	16.06	7.73	3.05%	1.01%	3006	1674
90	17.91	10.0	1.38%	0.98%	3375	2605
91	13.87	6.76	1.03%	0.48%	3058	2434

These examples demonstrate that as the amount of perlite was decreased (which increased the concentration of fiber), the tensile strength, elongation, and Young's modulus all increased, except after the amount of perlite was reduced below a certain amount. Both the tensile strength and the Young's modulus continued to increase until the perlite was left out altogether, as in Example 91. However, the ability of the material to elongate increased as the perlite was decreased, until less than 0.2 kg was used, after which the elongation dropped considerably. Reducing the amount of perlite beyond a certain point in this mix design results in an increased amount of defects in the sheets, which decreases the strength, elongation, and elasticity of the sheets.

In general, as the amount of perlite is decreased, the concentrations of fiber, rheology modifying agent, and hydraulic cement are increased, which would be expected to add to the tensile strength. In addition, increasing the concentration of cement would add to the stiffness (modulus) while negatively affecting the elongation ability of the product.

Another interesting point is that the ratio of tensile strength in the strong and weak directions was only about 2:1 in these sheets, whereas in paper products the ratio is typically 3:1.

While the sheets tested above were substantially dry, sheets made according to Examples 86-91 were further dried in an oven in order to obtain a sheet of maximum dryness. The further drying of the sheets was performed in order to portray a more accurate picture of the strength and other properties of the sheets under constant conditions. Depending on the mix designs, humidity during the test procedures, or other variables, the sheets would be expected to absorb or retain a certain amount of moisture. The strength, elongation, and modulus of elasticity results for the further dried sheets are set forth as follows:

Example	Strength (MPa) 0°	Strength (MPa) 90°	Elongation ($\Delta L/L$) 0°	Elongation ($\Delta L/L$) 90°	Modulus (MPa) 0°	Modulus (MPa) 90°
86	14.01	N/A	1.53%	N/A	2559	N/A
87	13.6	6.23	1.34%	1%	1799	2071
88	16.81	8.11	1.76%	1.08%	2659	1587
89	19.32	8.91	1.82%	1.16%	4002	1609
90	20.25	11.23	1.41%	0.63%	3448	1536
91	17.5	N/A	0.81%	N/A	3457	N/A

As shown by these examples, totally drying the sheets decreases the elongation somewhat, whereas the strength and modulus increased. These examples therefore teach that where increased strength and stiffness are important, the sheet should be totally dry. Where increased elongation is important, the elongation may be controlled with the humidity of the sheet.

Examples 92-96

Hydraulically settable sheets having a thickness of 0.4 mm were manufactured according to the process set forth in Examples 37-52 from a hydraulically settable mixture containing the following components:

Portland Cement	1.0 kg
$CaCO_3$	variable
Tylose® FL 15002	0.20 kg
Fiber (Southern pine)	0.25 kg
Water	variable

The effect of adding varying amounts of calcium carbonate was studied to determine the effect on the properties of the material, particularly the strength properties of the hardened sheet. Because of the reduced water-absorbing behavior of calcium carbonate compared to perlite, it was not necessary to decrease the amount of water by the same level as the amount of calcium carbonate was decreased in order to maintain the same level of rheology and workability.

Example	$CaCO_3$	Water
92	5.0 kg	2.25 kg
93	4.0 kg	2.15 kg
94	3.0 kg	2.05 kg
95	2.0 kg	2.00 kg
96	1.0 kg	1.96 kg

The strength, elongation, and Young's modulus of each of the totally dry sheets formed from the different mix designs are set forth as follows:

Example	Strength (MPa) 0°	90°	Elongation ($\Delta L/L$) 0°	90°	Modulus (MPa) 0°	90°
92	11.59	N/A	N/A	N/A	N/A	N/A
93	16.16	N/A	0.72%	N/A	4638	N/A
94	14.82	5.22	0.97%	0.42%	4521	3521
95	20.43	8.26	1.11%	0.56%	4301	2773
96	18.43	7.98	1.13%	0.51%	3902	3320

The use of calcium carbonate yields sheets with a smoother, more defect-free surface as well as a more homogeneous microstructure compared to where perlite is used.

Examples 97-103

Hydraulically settable sheets having a thickness of 0.4 mm were manufactured according to the process set forth in Examples 37-52 from a hydraulically settable mixture containing the following components:

Portland Cement	1.0 kg
Perlite	0.5 kg
Mica	0.5 kg
Tylose® FL 15002	variable
Fiber (Southern pine)	0.25 kg
Water	variable

The level of Tylose® was altered in order to determine the effect of increasing amounts of Tylose® within the hydraulically settable mixture. Increasing the amount of

Tylose® within the mixture required the addition of more water in order to dissolve the Tylose® and maintain similar rheology and workability.

Example	Tylose®	Water
97	0.1 kg	2.25 kg
98	0.3 kg	2.75 kg
99	0.4 kg	3.00 kg
100	0.5 kg	3.25 kg
101	0.6 kg	3.50 kg
102	0.7 kg	3.75 kg
103	0.8 kg	4.0 kg

The tensile strength and elongation properties increased up to a point as more Tylose® was added, while the Young's modulus fluctuated. The results of testing oven dried sheets made using the various mix designs are as follows:

Example	Strength (MPa)		Elongation (ΔL/L)		Modulus (MPa)	
	0°	90°	0°	90°	0°	90°
97	N/A	N/A	N/A	N/A	N/A	N/A
98	13.84	7.25	1.41%	0.75%	2954	1692
99	16.43	7.9	1.9%	0.83%	2400	2075
100	21.31	11.58	3.64%	1.06%	3347	2370
101	16.11	10.35	1.84%	1.13%	2816	1797
102	15.73	9.56	1.81%	0.93%	2690	1851
103	18.86	10.33	2.35%	1.45%	2790	1570

As illustrated, increasing the concentration of Tylose® will generally tend to increase the tensile strength, modulus, and elongation before rupture. A higher elongation ability would be expected to aid in curling the rim of a cup formed from a sheet, while increasing the strength of the sheet at a score cut. However, as the concentration of Tylose® is increased above a certain amount, the material becomes less workable and more defects are introduced within the structural matrix, which would be expected to reduce the strength, modulus, and elongation of the sheet. Nevertheless, the amount of defects (and resulting strength properties) can be improved by optimizing the calendering process.

Example 104

Based on the understanding that tensile strength and elongation generally increase as both the amount of fiber and Tylose® is increased within a mix design, a mix design was made which maximized both. The cementitious mixture included the following components:

Portland cement	1.0 kg
Water	2.2 kg
Perlite	0.1 kg
Fiber (Southern pine)	0.25 kg
Tylose® FL 15002	0.5 kg

The mixture was extruded and then passed between a series of pairs of rollers into a sheet having a thickness of 0.4 mm. The totally dried sheet was found to have superior strength and elongation properties. The tensile strength was 39.05 MPa in the strong direction and 18.86 MPa in the weak direction; the elongation was 1.97% in the strong direction and 1.23% in the weak direction; and the modulus of elasticity was 3935 in the strong direction and 2297 in the weak direction, which is comparable to normal paper.

Examples 105-109

Hydraulically settable sheets having a thickness of 0.4 mm were manufactured according to the process set forth in Examples 37-52 from a hydraulically settable mixture containing the following components:

Portland Cement	1.0 kg
Hollow glass spheres (4000 psi)	variable
Tylose® FL 15002	0.2 kg
Fiber (Southern pine)	0.25 kg
Water	variable

The effect of adding varying amounts of hollow glass spheres was studied to determine the effect on the properties of the material, particularly the strength properties of the hardened sheet. Although glass spheres do not absorb large amounts of water, less water was required to maintain the same rheology as the amount of glass spheres was decreased because of the corresponding decrease in interparticulate space.

Example	Glass Spheres	Water
105	0.5 kg	1.6 kg
106	0.4 kg	1.45 kg
107	0.3 kg	1.40 kg
108	0.2 kg	1.35 kg
109	0.1 kg	1.25 kg

The strength, elongation, and Young's modulus of each of the totally dry sheets formed from the different mix designs are set forth as follows:

Example	Strength (MPa)		Elongation ($\Delta L/L$)		Modulus (MPa)	
	0°	90°	0°	90°	0°	90°
105	10.34	3.69	2.2%	1.52%	1166	620
106	11.1	4.79	2.02%	1.49%	1446	677
107	12.38	5.71	1.58%	1.15%	1800	870
108	14.52	6.89	1.5%	1.1%	1935	1220
109	19.45	9.66	1.54%	0.96%	2660	1741

As seen with glass spheres, the modulus of elasticity is much lower while the elongation is fairly high compared to other mix designs. The sheets are therefore more pliable and elastic. The sheets formed in Examples 105-109 were highly thermally insulating, with k-factors ranging from 0.08-0.14 W/m•K.

Examples 110-113

Hydraulically settable sheets having a thickness of 0.4 mm were manufactured according to the process set forth in Examples 37-52 from a hydraulically settable mixture containing the following components:

Portland Cement	1.0 kg
Perlite	0.5 kg
Mica	variable
Tylose® FL 15002	0.2 kg
Fiber (Southern pine)	0.25 kg
Water	variable

The effect of adding varying amounts of mica was studied to determine the effect on the properties of the material, particularly the strength properties of the hardened sheet. Because of the water-absorbing behavior of mica, it was necessary to increase the amount of water as the amount of mica was increased in order to maintain the same level of rheology and workability.

Example	Mica	Water
110	1.0 kg	2.7 kg
111	1.5 kg	2.9 kg
112	2.0 kg	3.0 kg
113	2.5 kg	3.2 kg

The strength, elongation, and Young's modulus of each of the totally dry sheets formed from the different mix designs are set forth as follows:

Example	Strength (MPa)		Elongation ($\Delta L/L$)		Modulus (MPa)	
	0°	90°	0°	90°	0°	90°
110	9.92	4.61	0.825%	0.652%	2127	1257
111	9.37	5.3	0.71%	0.49%	3079	2188
112	11.14	4.05	0.79%	0.314%	3100	1520
113	11.41	4.76	0.58%	0.32%	2693	1282

Increasing the concentration of mica increases the strength of the sheets while reducing their elongation ability. Sheets containing larger amounts of mica became very brittle.

Example 114

Using any of the mix designs set forth above, a hydraulically settable mixture is made by substituting gypsum hemihydrate for the hydraulic cement in roughly the same quantity by weight. The hydraulically settable mixture will have a faster setting time but will generally result in sheets having similar strength, elongation, and stiffness properties.

Example 115

Using any of the mix designs set forth above, a hydraulically settable mixture is made by substituting calcium oxide for the hydraulic cement. The hydraulically settable mixture will have a slower setting time due to the slower reaction between calcium oxide

and carbon dioxide, but will generally result in sheets having similar strength, elongation, and stiffness properties. However, by removing much of the water within the mixture during or after the molding process, the desired level of quickly attained green strength will be possible.

Example 116

A hydraulically settable mixture is made having the following components:

Gypsum hemihydrate	1.0 kg
Perlite	0.5 kg
Tylose®	0.075 kg
Fiber	0.25 kg
Water	2.6 kg

The gypsum, Tylose®, fiber, and water are mixed together in a high shear mixer for 3 minutes, after which the perlite is added and mixed in a low shear mixer for an additional 3 minutes.

The mixture is extruded into a sheet having a thickness of 6 mm and then calendered in order to reduce the thickness of the sheets in steps to yield sheets having a final thickness ranging between 0.25 mm to 0.5 mm.

These sheets are readily formed into an appropriate food or beverage container using any appropriate procedure set forth in this specification. The strength properties are comparable to containers made using hydraulic cement and may be useful in the place of, *e.g.*, paper, cardboard, or polystyrene containers.

Example 117

Any of the cementitious mix designs using hydraulic cement in the above examples is altered to include about 25% gypsum hemihydrate by weight of the hydraulic cement. The gypsum acts as a water absorbing component (or internal drying agent) and results in quicker form stability. The strength properties of containers formed therefrom are comparable to mixtures not including gypsum.

Example 118

A set accelerator is included within any of the above mix designs, resulting in a hydraulically settable mixture that will more quickly achieve form stability. The final

strength of the material will be comparable to materials in which a set accelerator is not used.

Example 119

Waste hydraulically settable containers were composted along with waste food. After 4 weeks, the containers were completely broken down and resulted in compost which substantially resembled potting soil.

Examples 120-123

The cementitious mixture of Example 4 was altered by adding varying amounts of abaca fiber as follows:

Example	Abaca Fiber
120	200 g
121	250 g
122	300 g
123	350 g

The resultant percentages by weight of the added abaca fiber in Examples 120-123 were 5.3%, 6.6%, 7.8%, and 9.0%. The cementitious mixtures were extruded and then passed between a pair of rollers to form thin sheets having a thickness of about 0.2 mm to 2 mm. As more fiber was added, the cured cementitious material had greater flexibility and toughness, which made it more suitable for making a fold or bend in the material. The added fibers make the cured sheets suitable to make a hinge, such as by scoring the sheets with single, double or multiple scores, which allow the sheets to be bent.

Examples 124-127

The cementitious mixtures of Examples 124-127 were made which contained the following components:

Example	Cement	Water	Tylose®	Abaca Fiber
124	0.75 kg	2.0 kg	100 g	260 g
125	1.5 kg	2.0 kg	100 g	260 g
126	3.0 kg	2.0 kg	100 g	260 g
127	6.0 kg	2.0 kg	100 g	260 g

The cementitious mixtures were mixed in a high energy mixer for 5 minutes, extruded, and then passed through a pair of rollers to form sheets having thicknesses ranging from about 0.2 mm to 1.5 mm. Tylose® FL 15002 was used. As less cement was included, the effective amount of fiber increased, making the final product more ductile, tough, and less brittle.

The cementitious mixture of Example 125 was rolled onto a spool much like paper and could be used thereafter much like paper. For example, the rolled paper was later formed into a variety of objects such as a box, french fry carton, etc. In order to fold the sheet it was preferable to score it first and then fold the sheet along the score.

Examples 128-129

The cementitious mixture of Example 125 was altered to instead contain either 150 g or 200 g of Tylose® FL 15002. By adding more Tylose®, the resulting cementitious mixture had more plasticity. The mixtures of Examples 128 and 129 were molded and shaped into the same objects as those in Example 125.

Example 130

A cementitious mixture was prepared which had the following components:

Portland Cement	1.0 kg
Water	2.3 kg
Tylose® FL 15002	200 g
Abaca Fiber	250 g
Perlite	1.1 kg

The cement, water, Tylose®, and abaca fiber were mixed together in a high speed, high shear mixer for five minutes. Thereafter, the perlite was added to the mixture, which was mixed under low speed mixing conditions for five minutes. The cementitious mixture was made into thin sheets of about 1 mm or less by first extruding, and then passing through a pair of rollers, the cementitious mixture. Some of the thin sheets were formed into the shape of a box before hardening.

Other sheets were scored to form a hinge and allowed to cure before they were formed into the shape of a box. The sheets were readily bent along the line where the sheet was scored. The walls of the box were glued together using adhesion methods known to those skilled in the art. Where the walls were not glued together, such as in the case of a flap or other enclosure means, the fiber provided adequate strength properties so that the flaps could repeatedly bend along the hinge without breaking.

Example 131

The cementitious mixture of Example 130 was altered to contain 500 g of abaca fiber and formed into the same objects as in Example 130. The final cured product of this example had greater fracture energy, toughness, and tensile strength. In addition, the hinges formed in the containers were more durable and were better able to resist cracking or fracturing due to the stresses applied in bending the flaps along the hinge.

The present invention may be embodied in other specific forms without departing from its spirit or essential characteristics. The described embodiments are to be considered in all respects only as illustrative and not restrictive. The scope of the present invention is, therefore, indicated by the appended claims rather than by the foregoing description. All changes which come within the meaning and range of equivalency of the claims are to be embraced within their scope.

What is claimed is:

1. An apparatus comprising a hinge formed of a hydraulically settable matrix and a fibrous material within the hydraulically settable matrix at the hinge location, wherein the hydraulically settable matrix comprises the chemical reaction products of a hydraulically settable binder and water.

2. The apparatus of claim 1, wherein said hinge is a living hinge.

3. The apparatus of claim 1, wherein said hinge is a nonliving hinge.

4. The apparatus of claim 1, wherein said hydraulically settable matrix of said hinge has a thickness of about 0.01 to 1 millimeter.

5. The apparatus of claim 1, wherein said hydraulically settable matrix of said hinge has a thickness of about 0.05 to 0.5 millimeter.

6. The apparatus of claim 1, wherein said hydraulically settable binder is a hydraulic cement.

7. The apparatus of claim 6, wherein said hydraulic cement is selected from the group consisting of portland cement, microfine cement, slag cement, calcium aluminate cement, plaster, silicate cement, gypsum cement, phosphate cement, high-alumina cement, magnesium oxychloride cement, aggregates coated with microfine cement particles, and mixtures thereof.

8. The apparatus of claim 1, wherein said hydraulically settable binder is included in an amount from about 1 percent to 70 percent by volume of the total solids of the hydraulically settable mixture.

9. The apparatus of claim 1, wherein said hydraulically settable mixture has a water to hydraulically settable binder ratio in the range of about 0.1:1 to 10:1.

10. The apparatus of claim 1, wherein said fibrous material is selected from the group consisting of glass, cellulose, hemp, abaca, cotton, metal, ceramic, silica, wood pulp, paper pulp, carbon, synthetic polymers, and mixtures thereof.

11. The apparatus of claim 1, wherein said fibrous material comprises an inorganic precipitate formed in situ in said hydraulically settable binder.

12. The apparatus of claim 1, wherein said fibrous material comprises individual fibers having an aspect ratio of at least about 10:1.

13. The apparatus of claim 1, wherein said fibrous material comprises individual fibers having an aspect ratio of at least about 100:1.

14. The apparatus of claim 1, wherein said fibrous material comprises individual fibers having an aspect ratio from about 200:1 to 300:1.

15. The apparatus of claim 1, wherein said fibrous material comprises individual fibers having a length that is at least about 10 times the average diameter of the individual particles of the hydraulically settable binder.

16. The apparatus of claim 1, wherein said fibrous material comprises individual fibers having a length that is at least about 100 times the average diameter of the individual particles of the hydraulically settable binder.

17. The apparatus of claim 1, wherein said fibrous material comprises individual fibers having a length that is at least about 1,000 times the average diameter of the individual particles of the hydraulically settable binder.

18. The apparatus of claim 1, wherein said fibrous material is included in an amount from about 0.2 percent to 50 percent by volume of the total solids of the hydraulically settable mixture.

19. The apparatus of claim 1, wherein said fibrous material is included in an amount from about 1 percent to 30 percent by volume of the total solids of the hydraulically settable mixture.

20. The apparatus of claim 1, wherein said fibrous material is included in an amount from about 5 percent to 15 percent by volume of the total solids of the hydraulically settable mixture.

21. The apparatus of claim 1, wherein said hydraulically settable matrix further comprises a rheology-modifying agent.

22. The apparatus of claim 21, wherein said rheology-modifying agent comprises a polysaccharide or any derivative thereof.

23. The apparatus of claim 21, wherein said rheology-modifying agent comprises a cellulose-based material, including cellulose and any derivative thereof.

24. The apparatus of claim 23, wherein said cellulose-based material is selected from the group consisting of methylhydroxyethylcellulose, hydroxymethylethylcellulose, methylcellulose, hydroxyethylcellulose, carboxymethylcellulose, ethylcellulose, hydroxyethylpropylcellulose, and mixtures thereof.

25. The apparatus of claim 23, wherein said cellulose-based material comprises methylhydroxyethylcellulose.

26. The apparatus of claim 22, wherein said polysaccharide comprises a starch-based material, including starches and any derivative thereof.

27. The apparatus of claim 26, wherein said starch-based material is selected from the group consisting of amylopectin, amylose, sea-gel, starch acetates, starch hydroxyethyl ethers, ionic starches, long-chain alkyl starches, dextrins, amine starches, phosphate starches, dialdehyde starches, and mixtures thereof.

28. The apparatus of claim 22, wherein said polysaccharide is selected from the group consisting of alginic acid, phycocolloids, agar, gum arabic, guar gum. locust bean gum, gum karaya, gum tragacanth, and mixtures thereof.

29. The apparatus of claim 21, wherein said rheology-modifying agent comprises a protein-based material, including proteins and any derivative thereof.

30. The apparatus of claim 29, wherein said protein-based material is selected from the group consisting of Zein®, gelatin, glue, casein, and mixtures thereof.

31. The apparatus of claim 21, wherein said rheology-modifying agent is selected from the group consisting of cellulose-based materials; starch-based materials, protein-based materials, and mixtures thereof.

32. The apparatus of claim 21, wherein said rheology-modifying agent is a synthetic material selected from the group consisting of polyvinyl alcohol, polyvinyl pyrrolidone, polyvinyl methylether, polyacrylic acids, polyacrylic acid salts, polyvinylacrylic acids, polyvinylacrylic acid salts, polyacrylimides, ethylene oxide polymers, and mixtures thereof.

33. The apparatus of claim 21, wherein said rheology-modifying agent is selected from the group consisting of synthetic clay and latex.

34. The apparatus of claim 21, wherein said rheology-modifying agent is included in an amount from about 0.1 percent to 30 percent by volume of the total solids of the hydraulically settable mixture.

35. The apparatus of claim 21, wherein said rheology-modifying agent is included in an amount from about 0.5 percent to 15 percent by volume of the total solids of the hydraulically settable mixture.

36. The apparatus of claim 21, wherein said rheology-modifying agent is included in an amount from about 1 percent to 10 percent by volume of the total solids of the hydraulically settable mixture.

37. The apparatus of claim 1, wherein said hydraulically settable matrix further comprises an aggregate material.

38. The apparatus of claim 37, wherein said aggregate material is selected from the group consisting of metals, ceramics, clay, rock, silica, and mixtures thereof.

39. The apparatus of claim 37, wherein said aggregate material is selected from the group consisting of alumina, perlite, vermiculite, exfoliated rock, hollow glass spheres, sodium silicate macrospheres, lightweight concrete, porous ceramic spheres,

aerogel, lightweight expanded clay, expanded fly ash, expanded slag, pumice, sand, gravel, limestone, sandstone, gypsum, ground quartz, and mixtures thereof.

40. The apparatus of claim 37, wherein said aggregate material is selected from the group consisting of cork, seeds, granulated starches, gelatins, solid agar-type materials, and mixtures thereof.

41. The apparatus of claim 37, wherein said aggregate material comprises a biodegradable polymeric material.

42. The apparatus of claim 37, wherein said aggregate material is included in an amount up to about 90 percent by volume of the total solids of the hydraulically settable mixture.

43. The apparatus of claim 37, wherein said aggregate material is included in an amount from about 5 percent to 70 percent by volume of the total solids of the hydraulically settable mixture.

44. The apparatus of claim 37, wherein said aggregate material is included in an amount from about 20 percent to 50 percent by volume of the total solids of the hydraulically settable mixture.

45. The apparatus of claim 1, wherein said hydraulically settable matrix is flexible.

46. The apparatus of claim 1, wherein said hinge further comprises a pulp-containing material disposed thereon.

47. The apparatus of claim 46, wherein said pulp-containing material is a paper strip.

48. The apparatus of claim 1, further comprising a means for creating a discontinuous phase of finely dispersed, nonagglomerated voids within said hydraulically settable matrix.

49. The apparatus of claim 48, wherein said means for creating a discontinuous phase of voids comprises an air-entraining agent.

50. The apparatus of claim 49, wherein said air-entraining agent is a surfactant.

51. The apparatus of claim 49, wherein said air-entraining agent is selected from the group consisting of a polypeptide alkylene polyol, and a synthetic anionic biodegradable solution.

52. The apparatus of claim 49, further comprising a stabilizing agent for retaining the finely dispersed air voids within said hydraulically settable matrix.

53. The apparatus of claim 52, wherein said stabilizing agent is vinsol resin or a polysaccharide-based plasticizer.

54. The apparatus of claim 48, wherein said means for creating a discontinuous phase of nonagglomerated voids comprises a material which reacts with the components of the hydraulically settable matrix to produce a gas in order to incorporate voids into said hydraulically settable matrix.

55. The apparatus of claim 54, wherein said material which reacts with the components of said hydraulically settable matrix is a metal.

56. The apparatus of claim 55, wherein said metal is aluminum, zinc, or tin.

57. The apparatus of claim 55, further comprising a base which accelerates the production of gas.

58. The apparatus of claim 57, wherein said base is sodium hydroxide.

59. The apparatus of claim 1, further comprising a coating on at least a portion of the surface of the hydraulically settable matrix.

60. The apparatus of claim 59 wherein said coating is selected from the group consisting of melamine, polyvinyl chloride, polyvinyl alcohol, polyvinyl acetate, sodium silicate, calcium carbonate, polyacrylates, polyurethanes, clay, mica, wax, and ceramics.

61. An apparatus comprising:

(a) a first member;

(b) a second member adjacent to said first member;

and

(c) means for flexibly joining said first and second members so that said first and second members can be pivotally moved about said joining means relative to one another, wherein said joining means comprises a hydraulically settable matrix including the reaction products of a hydraulically settable binder and water.

62. The apparatus of claim 61, wherein said first and second members have a mechanical resistance to bending and elongation within a first range and wherein said joining means further comprises an area of reduced mechanical resistance to bending and elongation within a second range that is less than the first range of mechanical resistance.

63. The apparatus of claim 61, wherein said first and second members have a thickness within a first range and wherein said joining means further comprises an area of reduced thickness within a second range that is less than the first range of thickness.

64. The apparatus of claim 61, wherein said joining means further comprises a fibrous material dispersed within said hydraulically settable matrix.

65. The apparatus of claim 61, wherein each of said first and second members comprise a hydraulically settable matrix including the reaction products of a hydraulically settable binder and water.

66. The apparatus of claim 61, wherein said hydraulically settable matrix of said joining means has a thickness of about 0.01 to 1 millimeter.

67. The apparatus of claim 61, wherein said hydraulically settable binder is a hydraulic cement.

68. The apparatus of claim 64, wherein said fibrous material is selected from the group consisting of glass, cellulose, hemp, abaca, cotton, metal, ceramic, silica, wood pulp, paper pulp, carbon, synthetic polymers, and mixtures thereof.

69. The apparatus of claim 64, wherein said fibrous material comprises individual fibers having an aspect ratio of at least about 10:1.

70. The apparatus of claim 64, wherein said hydraulically settable matrix further comprises a rheology-modifying agent.

71. A container comprising:

(a) a first member;

(b) a second member adjacent to said first member; and

(c) means for flexibly joining said first and second members so that said first and second members can be pivotally moved about said joining means relative to one another between a first position wherein said first and second members are in straight alignment with one another and a plurality of other positions wherein said first and second members form an angle in relation to one another;

wherein said joining means and said first and second members comprise a hydraulically settable matrix, including the reaction products of a hydraulically settable binder and water, and a fibrous material dispersed in the hydraulically settable matrix.

72. The container of claim 71, wherein said first and second members have a mechanical resistance to bending and elongation within a first range and wherein said joining means further comprises an area of reduced mechanical resistance to bending and elongation within a second range that is less than the first range of mechanical resistance.

73. The container of claim 71, wherein said first and second members have a thickness within a first range and wherein said joining means further comprises an area of reduced thickness within a second range that is less than the first range of thickness.

74. The container of claim 71, wherein said hydraulically settable matrix of said joining means has a thickness of about 0.01 to 1 millimeter.

75. The container of claim 71, wherein said hydraulically settable binder is a hydraulic cement.

76. The container of claim 71, wherein said fibrous material is selected from the group consisting of glass, cellulose, hemp, abaca, cotton, metal, ceramic, silica, wood pulp, paper pulp, carbon, synthetic polymers, and mixtures thereof.

77. The container of claim 71, wherein said fibrous material comprises individual fibers having an aspect ratio of at least about 10:1.

78. The container of claim 71, wherein said hydraulically settable matrix further comprises a rheology-modifying agent.

79. An apparatus comprising a hinge having a cementitious structural matrix formed from a cementitious mixture comprising:

(a) about 1 to 70 percent by volume of a hydraulic cement;

(b) about 0.1 to 30 percent by volume of a rheology-modifying agent;

(c) a fibrous material comprising individual fibers having an aspect ratio of at least about 10:1;

(d) about 5 to 70 percent by volume of an aggregate material; and

(e) water added in an amount to result in a water to cement ratio of about 0.1:1 to 10:1;

wherein said cementitious structural matrix of said hinge has a thickness of about 0.01 to 1 millimeter.

80. The apparatus of claim 79, wherein said cementitious structural matrix of said hinge has a thickness of about 0.05 to 0.5 millimeter.

81. The apparatus of claim 79, wherein said hydraulic cement is a portland cement or a microfine cement.

82. The apparatus of claim 79, wherein said fibrous material is selected from the group consisting of glass fiber, cellulose fiber, metal fiber, ceramic fiber, silica fiber, carbon fiber, synthetic polymeric fiber, abaca fiber, and mixtures thereof.

83. The apparatus of claim 79, wherein said fibrous material is included in an amount from about 0.2 percent to 50 percent by volume of the cementitious mixture.

84. The apparatus of claim 79, wherein said rheology-modifying agent is selected from the group consisting of cellulose or derivatives thereof, starch or derivatives thereof, proteins or derivatives thereof, and mixtures thereof.

85. The apparatus of claim 79, wherein said rheology-modifying agent is selected from the group consisting of gums, a synthetic polymeric material, clay, and latex.

86. The apparatus of claim 79, wherein said rheology- modifying agent comprises methylhydroxyethylcellulose.

87. The apparatus of claim 79, wherein said aggregate material is selected from the group consisting of metals, ceramics, clay, rock, silica, hollow glass spheres, polymeric materials, and mixtures thereof.

88. The apparatus of claim 79, wherein said cementitious mixture further comprises an air entraining agent for creating a discontinuous phase of finely dispersed, nonagglomerated air voids within the cementitious mixture.

89. The apparatus of claim 88, wherein said air entraining agent is a surfactant.

90. The apparatus of claim 88, further comprising a stabilizing agent for retaining the finely dispersed air voids within the cementitious mixture as it cures into the cementitious structural matrix.

91. The apparatus of claim 79, wherein said cementitious mixture further comprises a metal which reacts with components of the cementitious mixture to produce a gas which is incorporated into the cementitious mixture as finely dispersed, nonagglomerated gas bubbles.

92. The apparatus of claim 91, wherein said metal is aluminum, zinc or tin.

93. The apparatus of claim 91, further comprising a base which accelerates the production of gas.

94. The apparatus of claim 93, wherein said base is sodium hydroxide.

95. The apparatus of claim 79, further comprising a coating on at least a portion of the surface of the cementitious structural matrix.

96. The apparatus of claim 79, wherein said hinge further comprises a pulp-containing material disposed thereon.

97. The apparatus of claim 96, wherein said pulp-containing material is a paper strip.

98. A method of making a hinge having a hydraulically settable matrix comprising the steps of:

(a) mixing a hydraulically settable binder, fibers, and water in order to form a hydraulically settable mixture;

(b) forming the hydraulically settable mixture into a form stable sheet of a predetermined thickness; and

(c) scoring said sheet to form a hinge in said hydraulically settable matrix.

99. The method of claim 98, wherein said structural matrix of said hinge has a thickness of about 0.01 to 0.5 millimeter.

100. The method of claim 98, wherein the step of forming the hydraulically settable mixture into a form stable sheet comprises extruding the hydraulically settable mixture through a die.

101. The method of claim 98, wherein the step of forming the hydraulically settable mixture into a form stable sheet comprises roller casting the hydraulically settable mixture.

102. The method of claim 98, further comprising the step of molding the sheet into a predetermined shape.

103. The method of claim 102, wherein the step of molding the sheet comprises hot pressing the sheet into a predetermined shape in a heated mold.

104. The method of claim 98, wherein the step of forming the hydraulically settable mixture into a form stable sheet is performed after the hydraulically settable mixture has been heated to a temperature of from about 50oC to 250oC.

105. A hydraulically settable sheet that has been scored to produce the hinge defined by claim 1.

106. A cementitious sheet that has been scored to produce the hinge defined by claim 79.

107. A cementitious container comprising at least one cementitious part that is flexibly attached to a second cementitious part with the hinge of claim 1.

108. The container of claim 107, wherein said container is in the shape of a clamshell container.

109. A method of manufacturing a bendable sheet having a hydraulically settable matrix, the method comprising the steps of:

(a) mixing a hydraulically settable binder, fibers, and water in order to form a hydraulically settable mixture;

(b) extruding said hydraulically settable mixture through a die;

(c) forming the extruded mixture into a form stable sheet of a predetermined thickness;

(d) hardening said sheet to a significant degree in an accelerated manner in order to quickly increase the yield stress of the hydraulically settable matrix; and

(e) cutting a score into a surface of said sheet that is substantially dried.

110. The method of claim 109, wherein said score defines a fold line along which said sheet may be bent.

111. A method of manufacturing a bendable sheet having a hydraulically settable matrix, the method comprising the steps of:

(a) mixing a hydraulically settable binder, fibers, and water in order to form a hydraulically settable mixture;

(b) extruding said hydraulically settable mixture through a die;

(c) forming the extruded mixture into a form stable sheet of a predetermined thickness;

(d) hardening said sheet to a significant degree in an accelerated manner in order to quickly increase the yield stress of the hydraulically settable matrix; and

(e) pressing a score into a surface of said sheet that is substantially dried.

112. The method of claim 111, wherein said score defines a fold line along which said sheet may be bent.

113. A method of manufacturing a bendable sheet having a hydraulically settable matrix, the method comprising the steps of:

(a) mixing a hydraulically settable binder, fibers, and water in order to form a hydraulically settable mixture;

(b) extruding said hydraulically settable mixture through a die;

(c) forming the extruded mixture into a form stable sheet of a predetermined thickness;

(d) hardening said sheet to a significant degree in an accelerated manner in order to quickly increase the yield stress of the hydraulically settable matrix; and

(e) cutting a perforation into said sheet that is substantially dried.

114. The method of claim 113, wherein said perforation defines a fold line along which said sheet may be bent.

AMENDED CLAIMS
[received by the International Bureau on 11 May 1995 (11.05.95); original claims 1-114 replaced by amended claims 1-42 (6 pages)]

1. An apparatus comprising a hinge formed of a hydraulically settable matrix and a fibrous material within the hydraulically settable matrix at the hinge location, wherein the hydraulically settable matrix comprises the chemical reaction products of a hydraulically settable binder and water.

2. The apparatus of claim 1, wherein said hydraulically settable matrix of said hinge has a thickness of about 0.01 to 1 millimeter.

3. The apparatus of claim 1, wherein said hydraulically settable binder is included in an amount from about 1 percent to 70 percent by volume of the total solids of the hydraulically settable matrix.

4. The apparatus of claim 1, wherein said fibrous material comprises individual fibers having an aspect ratio of at least about 10:1.

5. The apparatus of claim 1, wherein said hydraulically settable matrix further comprises a rheology-modifying agent.

6. The apparatus of claim 5, wherein said rheology-modifying agent is included in an amount from about 0.1 percent to 30 percent by volume of the total solids of the hydraulically settable matrix.

7. The apparatus of claim 1, wherein said hydraulically settable matrix further comprises an aggregate material.

8. The apparatus of claim 7, wherein said aggregate material is included in an amount up to about 90 percent by volume of the total solids of the hydraulically settable matrix.

9. An apparatus comprising a hinge having a cementitious structural matrix formed from a cementitious mixture comprising:

(a) about 1 to 70 percent by volume of a hydraulic cement;

(b) about 0.1 to 30 percent by volume of a rheology-modifying agent;

(c) a fibrous material comprising individual fibers having an aspect ratio of at least about 10:1;

(d) about 5 to 70 percent by volume of an aggregate material; and

(e) water added in an amount to result in a water to cement ratio of about 0.1:1 to 10:1;

wherein said cementitious structural matrix of said hinge has a thickness of about 0.01 to 1 millimeter.

10. The apparatus of claims 1 and 9, wherein said hinge is a living hinge.

11. The apparatus of claims 1and 9, wherein said hinge is a nonliving hinge.

12. The apparatus of claim 1, wherein said hydraulically settable binder is a hydraulic cement.

13. The apparatus of claims 9 and 12, wherein said hydraulic cement is selected from the group consisting of portland cement, microfine cement, slag cement, calcium aluminate cement, plaster, silicate cement, gypsum cement, phosphate cement, high-alumina cement, magnesium oxychloride cement, aggregates coated with microfine cement particles, and mixtures thereof.

14. The apparatus of claims 1 and 9, wherein said fibrous material is selected from the group consisting of glass, cellulose, hemp, abaca, cotton, metal, ceramic, silica, wood pulp, paper pulp, carbon, synthetic polymers, and mixtures thereof.

15. The apparatus of claims 1and 9, wherein said fibrous material is included in an amount from about 0.2 percent to 50 percent by volume of the total solids of the matrix.

16. The apparatus of claims 5 and 9, wherein said rheology-modifying agent comprises a polysaccharide or any derivative thereof.

17. The apparatus of claims 5 and 9, wherein said rheology-modifying agent is selected from the group consisting of cellulose-based materials, starch-based materials, protein-based materials, and mixtures thereof.

18. The apparatus of claims 5 and 9, wherein said rheology-modifying agent is a synthetic material selected from the group consisting of polyvinyl alcohol, polyvinyl pyrrolidone, polyvinyl methylether, polyacrylic acids, polyacrylic acid salts, polyvinylacrylic acids, polyvinylacrylic acid salts, polyacrylimides, ethylene oxide polymers, synthetic clay, latex, and mixtures thereof.

19. The apparatus of claims 7 and 9, wherein said aggregate material is selected from the group consisting of metals, ceramics, clay, rock, silica, and mixtures thereof.

20. The apparatus of claim 7 and 9, wherein said aggregate material is selected from the group consisting of alumina, perlite, vermiculite, exfoliated rock, hollow glass spheres, sodium silicate macrospheres, lightweight concrete, porous ceramic spheres, aerogel, lightweight expanded clay, expanded fly ash, expanded slag, pumice, sand, gravel, limestone, sandstone, gypsum, ground quartz, and mixtures thereof.

21. The apparatus of claims 1 and 9, wherein said matrix is flexible.

22. The apparatus of claims 1 and 9, wherein said hinge further comprises a pulp-containing material disposed thereon.

23. The apparatus of claim 22, wherein said pulp-containing material is a paper strip.

24. The apparatus of claims 1 and 9, further comprising a means for creating a discontinuous phase of finely dispersed, nonagglomerated voids within said matrix.

25. The apparatus of claim 24, wherein said means for creating a discontinuous phase of voids comprises an air-entraining agent.

26. The apparatus of claim 25, further comprising a stabilizing agent for retaining the finely dispersed voids within said matrix.

27. The apparatus of claim 24, wherein said means for creating a discontinuous phase of nonagglomerated voids comprises a material which reacts with

the components of said matrix to produce a gas in order to incorporate voids into said matrix.

28. The apparatus of claim 27, wherein said material which reacts with the components of said matrix is a metal.

29. The apparatus of claim 28, further comprising a base which accelerates the production of gas.

30. The apparatus of claims 1 and 9, further comprising a coating on at least a portion of a surface of said matrix.

31. An apparatus comprising:

(a) a first member;

(b) a second member adjacent to said first member; and

(c) means for flexibly joining said first and second members so that said first and second members can be pivotally moved about said joining means relative to one another, wherein said joining means comprises a hydraulically settable matrix including the reaction products of a hydraulically settable binder and water.

32. The apparatus of claim 31, wherein said first and second members have a mechanical resistance to bending and elongation within a first range and wherein said joining means further comprises an area of reduced mechanical resistance to bending and elongation within a second range that is less than the first range of mechanical resistance.

33. The apparatus of claim 31, wherein said first and second members have a thickness within a first range and wherein said joining means further comprises an area of reduced thickness within a second range that is less than the first range of thickness.

34. The apparatus of claim 31, wherein said joining means further comprises a fibrous material dispersed within said hydraulically settable matrix.

35. The apparatus of claim 31, wherein each of said first and second members comprise a hydraulically settable matrix including the reaction products of a hydraulically settable binder and water.

36. A container comprising:

(a) a first member;

(b) a second member adjacent to said first member; and

(c) means for flexibly joining said first and second members so that said first and second members can be pivotally moved about said joining means relative to one another between a first position wherein said first and second members are in straight alignment with one another and a plurality of other positions wherein said first and second members form an angle in relation to one another;

wherein said joining means and said first and second members comprise a hydraulically settable matrix, including the reaction products of a hydraulically settable binder and water, and a fibrous material dispersed in the hydraulically settable matrix.

37. A method of making a hinge having a hydraulically settable matrix comprising the steps of:

(a) mixing a hydraulically settable binder, fibers, and water in order to form a hydraulically settable mixture;

(b) forming the hydraulically settable mixture into a form stable sheet of a predetermined thickness; and

(c) scoring said sheet to form a hinge in said hydraulically settable matrix.

38. A hydraulically settable sheet that has been scored to produce the hinge defined by claim 1.

39. A cementitious container comprising at least one cementitious part that is flexibly attached to a second cementitious part with the hinge of claim 9.

40. The container of claim 39, wherein said container is in the shape of a clamshell container.

41. A method of manufacturing a bendable sheet having a hydraulically settable matrix, the method comprising the steps of:

(a) mixing a hydraulically settable binder, fibers, and water in order to form a hydraulically settable mixture;

(b) extruding the hydraulically settable mixture through a die;

(c) forming the extruded mixture into a form stable sheet of a predetermined thickness;

(d) hardening the sheet to a significant degree in an accelerated manner in order to quickly increase the yield stress of the hydraulically settable matrix; and

(e) cutting or pressing a score into a surface of the sheet, or cutting a perforation into the sheet that is substantially dried.

42. The method of claim 41, wherein said score or perforation defines a fold line along which said sheet may be bent.

STATEMENT UNDER ARTICLE 19

Please amend the claims in the above-identified application by cancelling sheets 103-116 of this application which contain the claims and abstract. Please substitute therefor attached sheets 103-109, which contain the new claims to be entered in this application. The abstract is identical to the abstract as originally filed and is found on substitute page 109.

The new claims submitted herewith cover substantially the same scope as the original claims, but have been amended to assist in examination in that the number of claims have been reduced. Accordingly, the numbering of the original claims does not correspond with the numbering of the new claims.

Applicants submit that the new claims submitted herewith accurately and distinctly point out the concepts applicants wish to claim. Applicants further submit that no new matter has been introduced by these new claims.

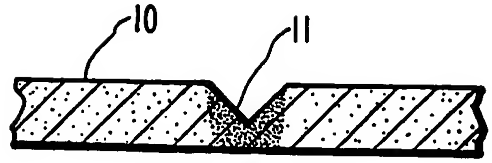

FIG. 1A

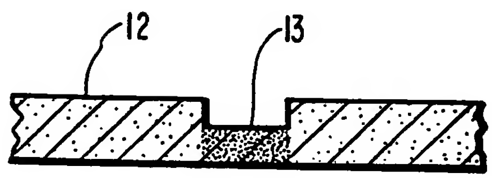

FIG. 1B

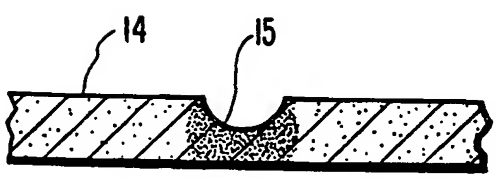

FIG. 1C

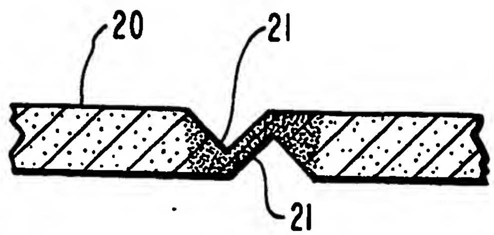

FIG. 2A

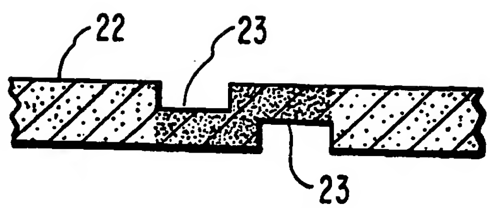

FIG. 2B

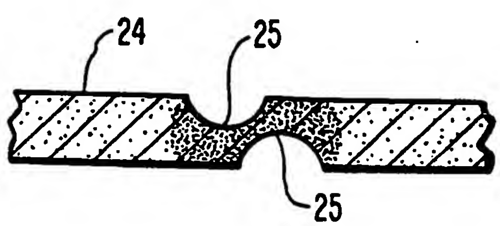

FIG. 2C

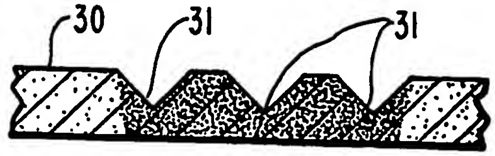

FIG. 3A

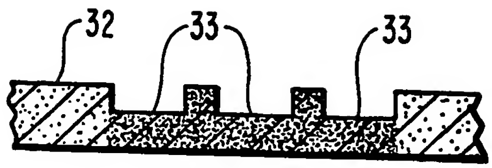

FIG. 3B

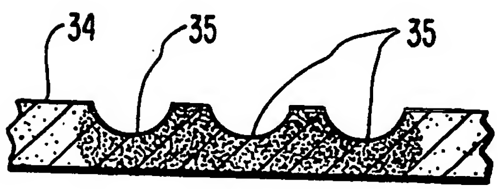

FIG. 3C

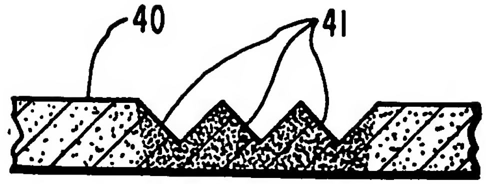

FIG. 3D

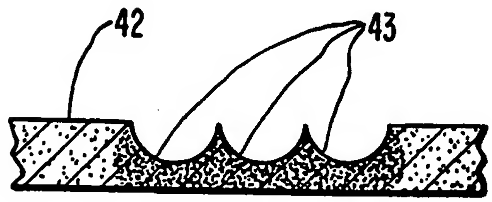

FIG. 3E

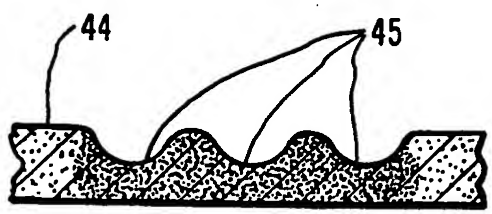

FIG. 3F

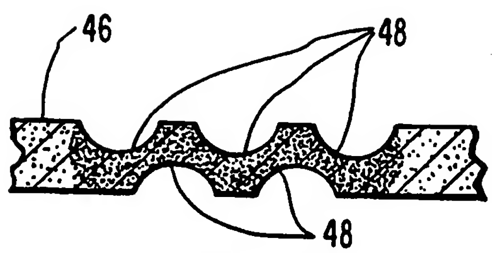

FIG. 3G

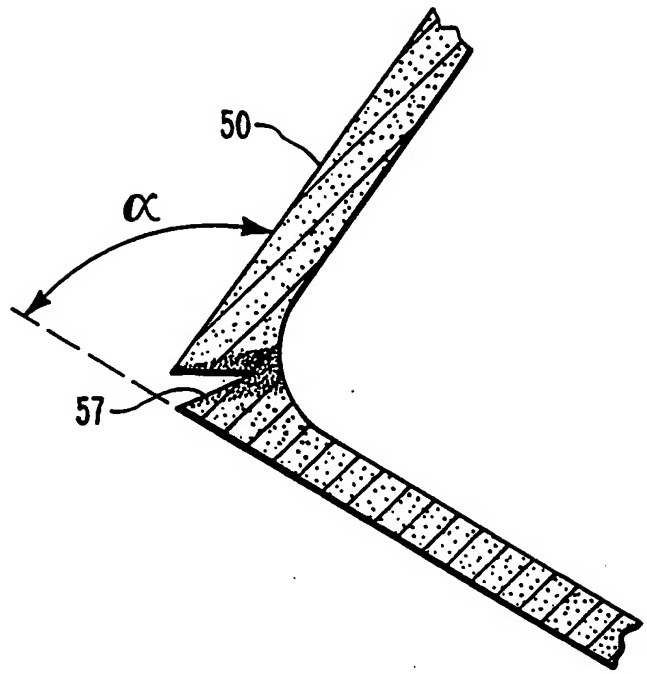

FIG. 4A

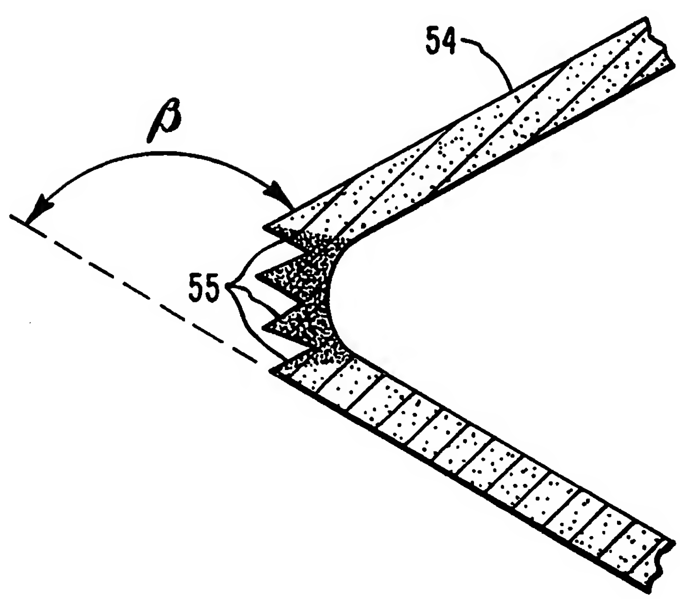

FIG. 4B

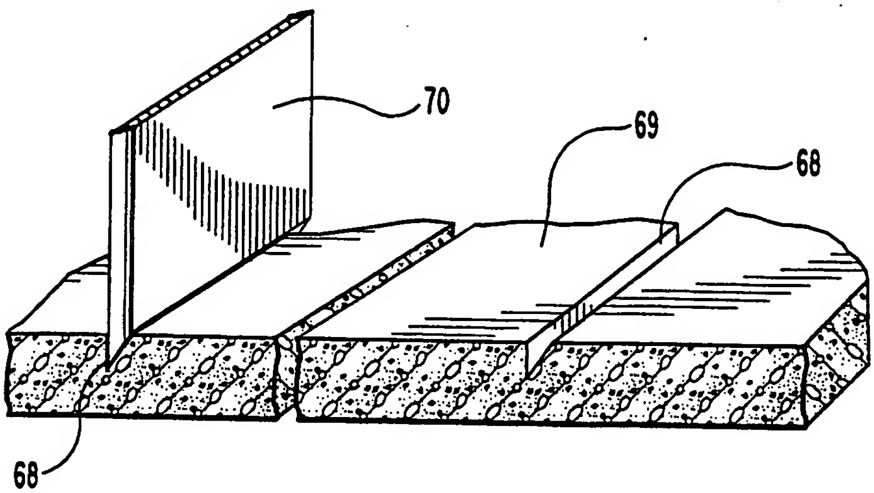

FIG. 5

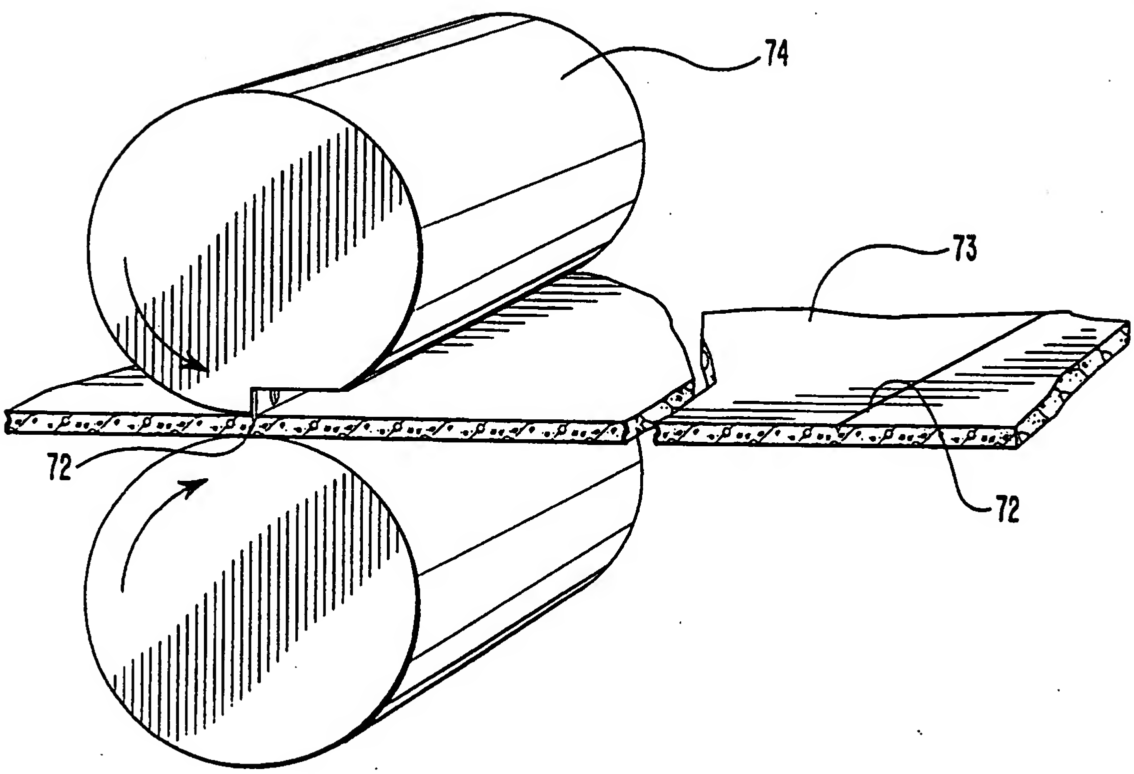

FIG. 6

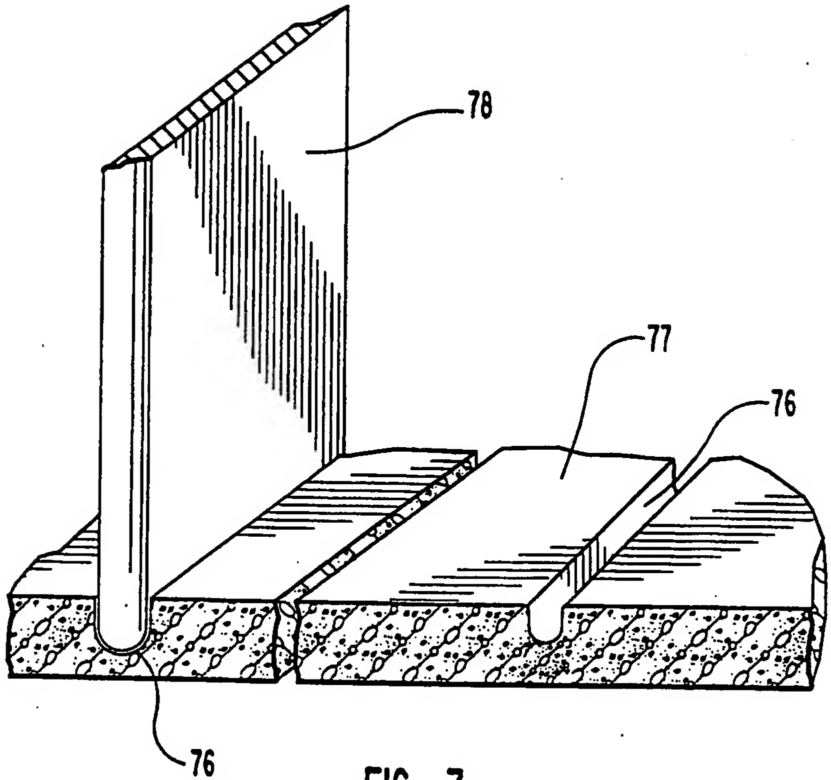

FIG. 7

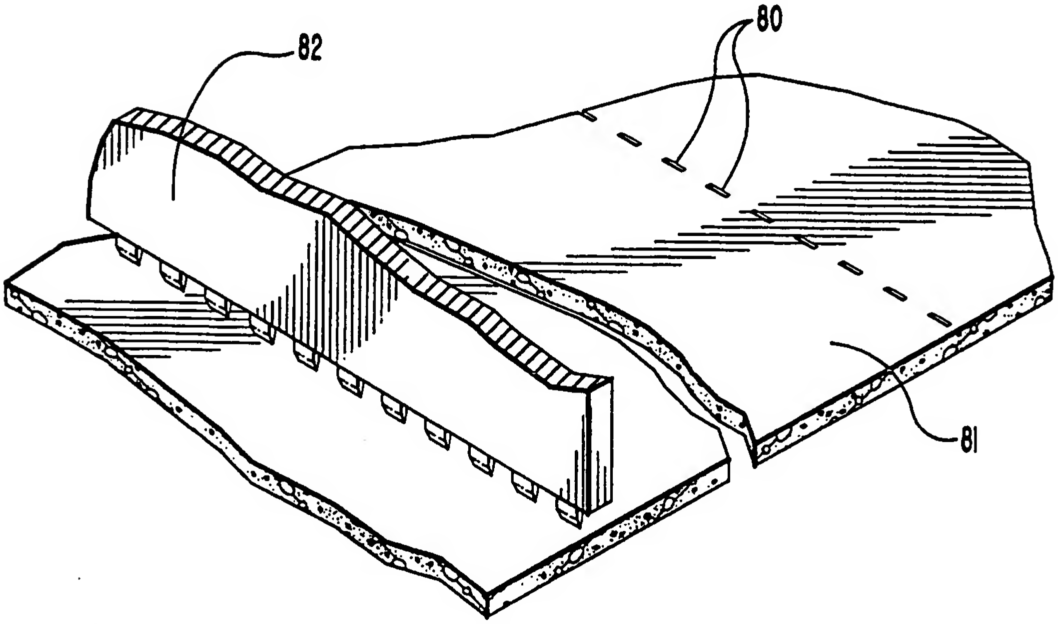

FIG. 8

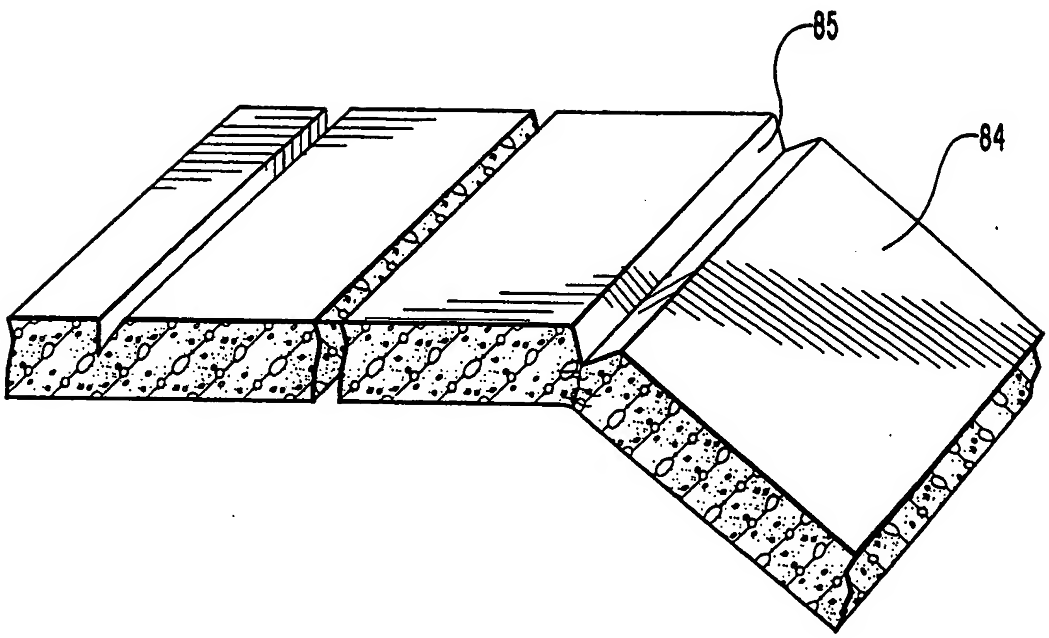

FIG. 9

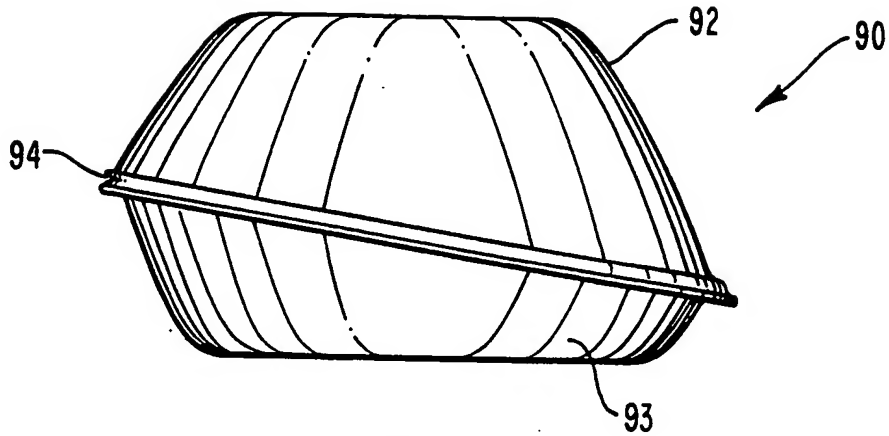

FIG. 10

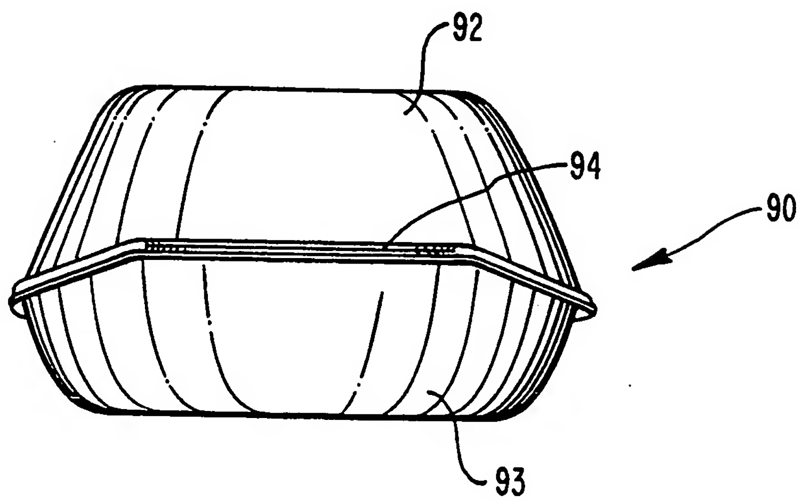

FIG. 11

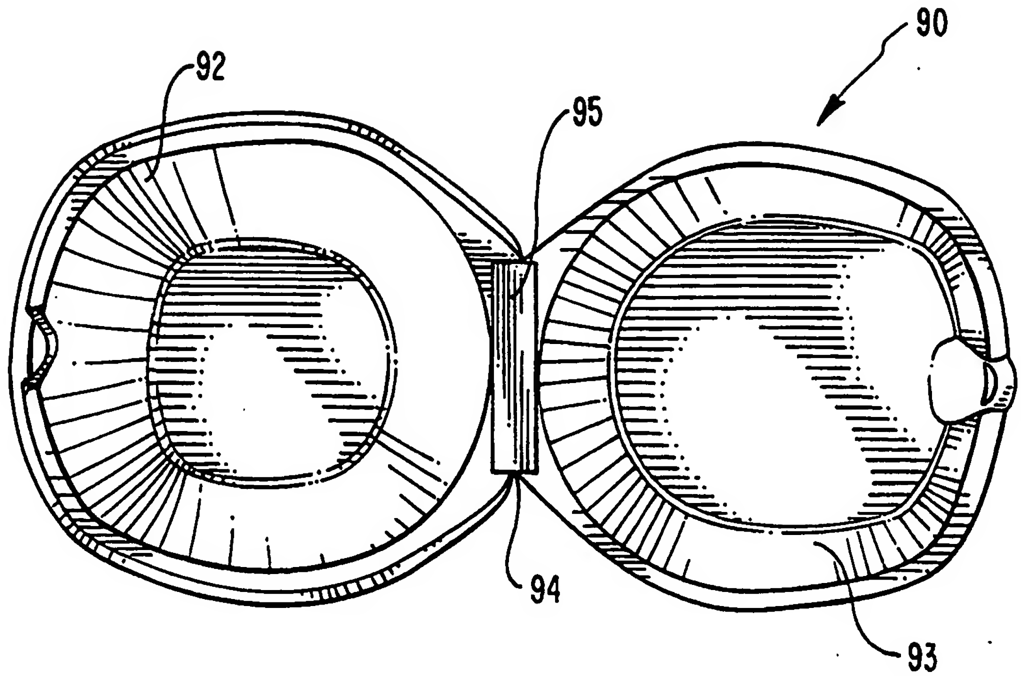

FIG. 12

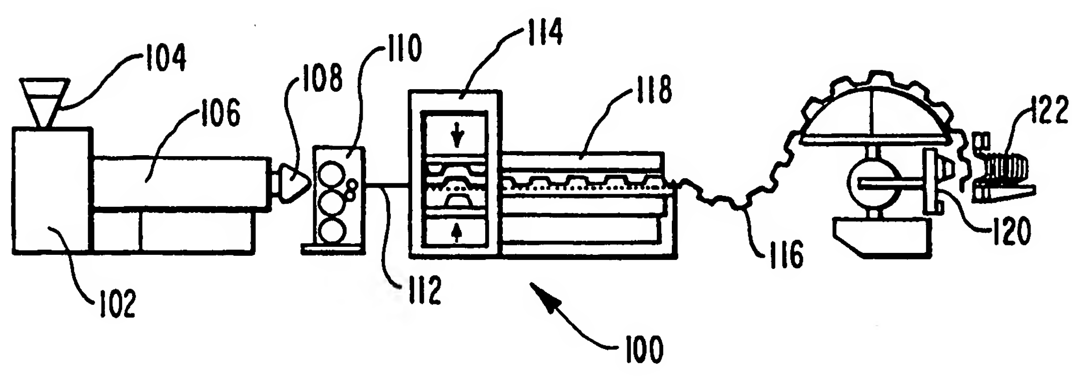

FIG. 13

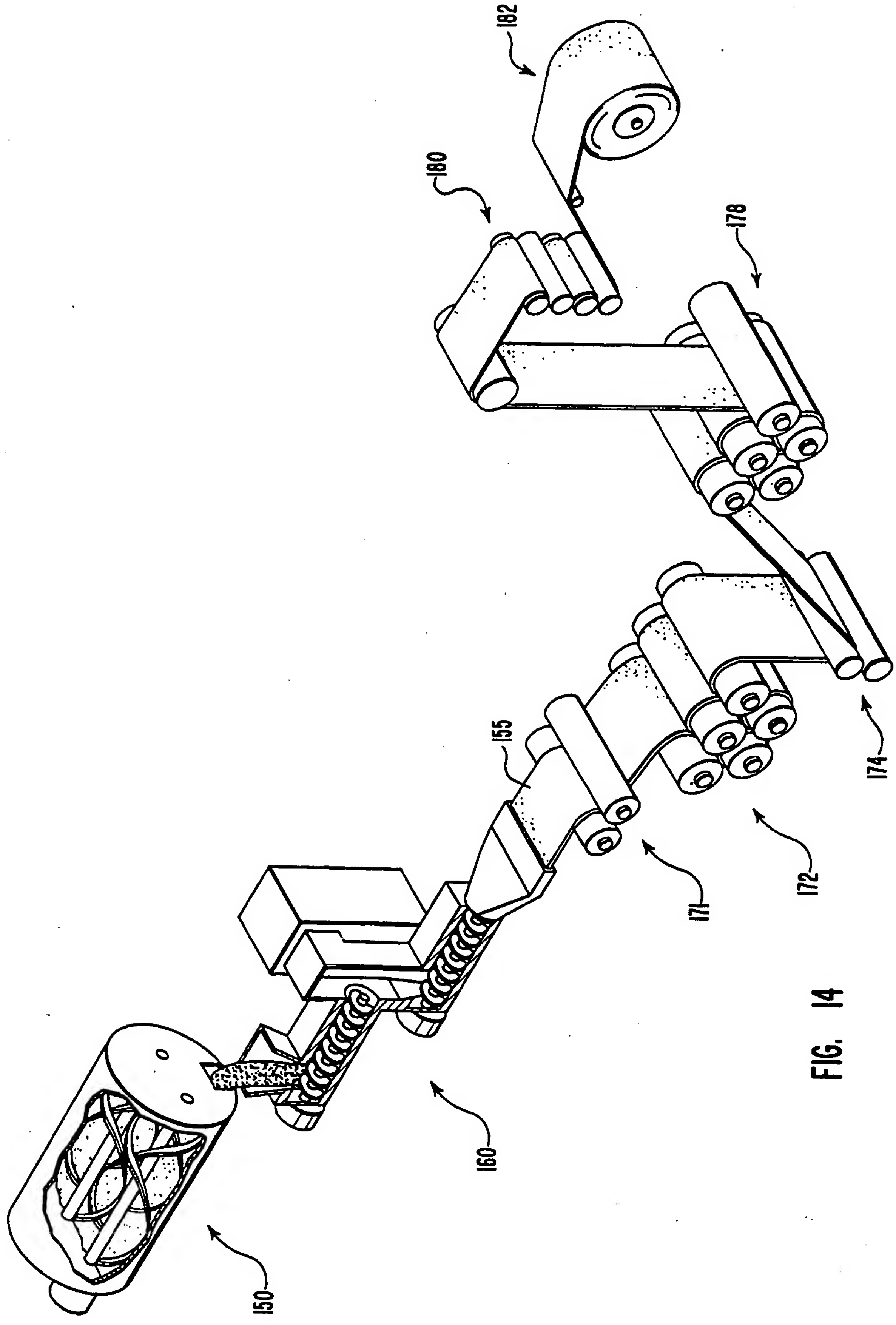

FIG. 14

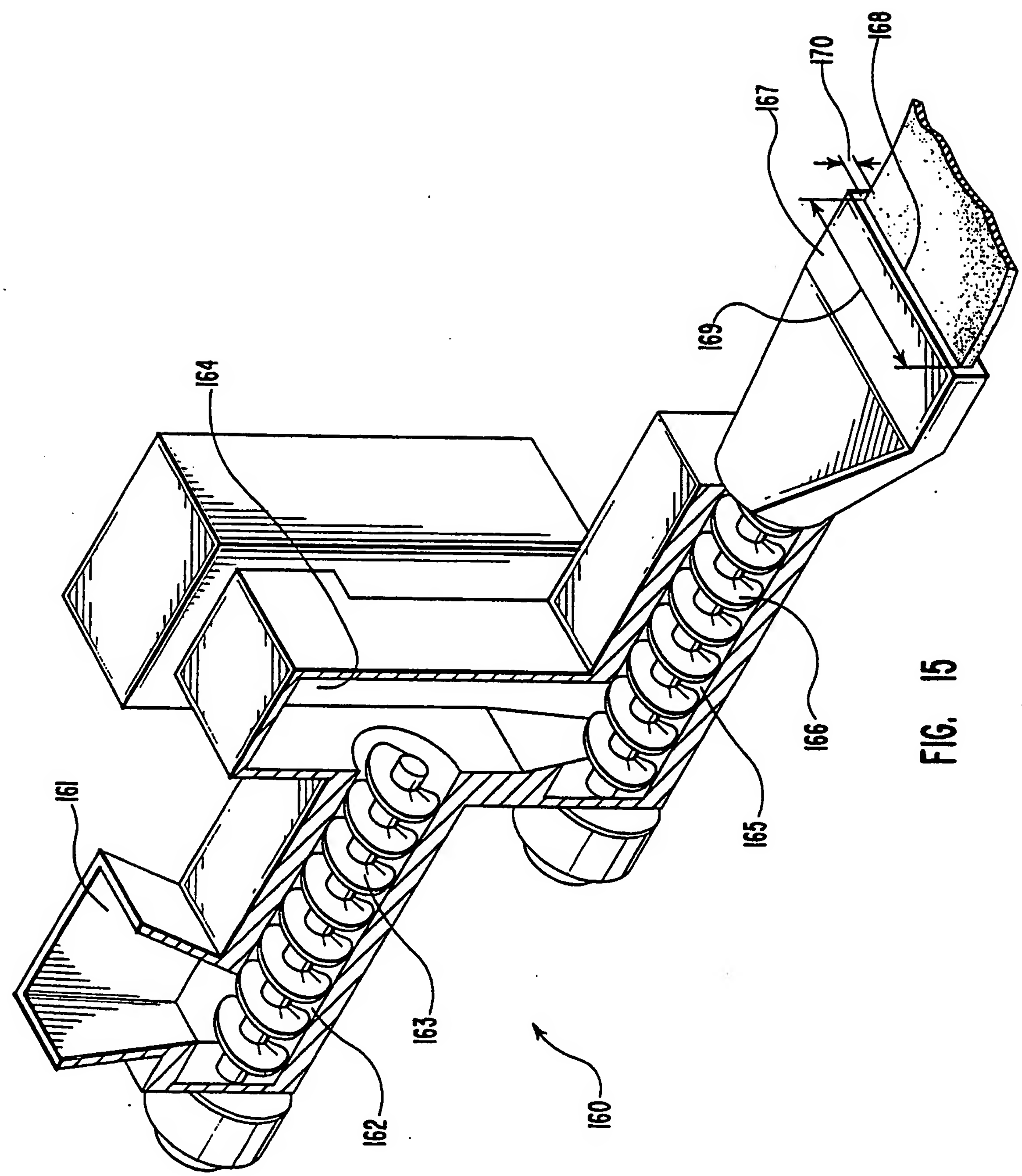

FIG. 15

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US94/14171

A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) :B32B 13/00; C04B 14/00; E05D 5/00
US CL :16/225, 385; 106/675, 729, 703; 264/296; 428/312.4, 703
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 16/225, 385; 106/675, 729, 703; 264/296; 428/312.4, 703

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

NONE

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

NONE

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X --- A	GB, A, 2 220 934 (REDLAND ROOF TILES LIMITED) 24 January 1990, see entire document.	1-3, 6-65, 67-73, 75-79, 81-14 -------------- 4, 5, 66, 74, 80

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be part of particular relevance

"E" earlier document published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
05 MARCH 1995	03 APR 1995

Name and mailing address of the ISA/US	Authorized officer
Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231	BLAINE R. COPENHEAVER